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An Improved Water Reclamation System Utilizing A Membrane Vapor Diffusion Still Concept

BY

W. B. COE and H. J. KOLNSBERG

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AN IMPROVED WATER RECLAMATION SYSTEM UTILIZING A MEMBRANE VAPOR
DIFFUSION STILL CONCEPT

by
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35321

SUMMARY

A laboratory model of an Improved Water Reclamation System based upon the membrane diffusion still concept was analyzed, designed and constructed. Its performance was demonstrated by means of a 116 hour test on a 1/5 scale system. The system achieved HS predicted processing rate and successfully recovered 98% of the available water. The water reclaimed from urine during the test was of potable quality throughout.

A conceptual design to determine prototype weight and thermal losses, based upon the test results achieved in this program, indicates an achievable launch weight per man of less than 7.5 lbs with an expendable weight of .0176 lbs per man day. Factors which influence this performance indicate the potentiality of the system for improvements in performance as the result of further development.

In order to obtain a high degree of reliability and further improve the system capability, additional effort is recommended to investigate membranes and membrane properties as applied to the membrane diffusion still concept.

INTRODUCTION

On space missions of extended duration, the weight of water required for human needs is in excess of the weight of systems required to recover water from the waste waters on board. One of the major sources of waste water is urine and the recovery of water from this source imposes very stringent requirements upon the potential recovery system.

In the past, systems employing a number of different concepts have been designed, constructed and tested to reclaim the water content of urine. Although some of these concepts have been successful in accomplishing their prime purpose, they all have had major disadvantages as space mission-oriented systems. Objections to their use center on their excessive weight, (including expendables) excessive power consumption, poor reliability or the inability to recover all of the available water from the urine.

This program was conducted to design, build and demonstrate an improved water reclamation system based on the concept of a membrane vapor diffusion still. The feasibility of this concept had been established by tests at Hamilton Standard prior to this program.

The concept offers a system which has only one moving part and is adaptable to zero-g operation with no additional moving parts. It requires only negligible electrical power and is capable of recovering nearly 100% of the available water contained in urine. It has a basically low fixed weight and a minimum requirement for expendables. The management of urine feed, water collection, and waste removal is accomplished simply and cleanly with no need for supplementary equipment. It can be designed for high packaging density and is capable of being scaled for different capacities without any major penalties.

1.0 **OBJECTIVES****1.1** **Scope of Work**

The major purpose of this program was to design, fabricate and develop an improved system for the reclamation of potable water from urine by means of phase change technique.

Some generalized properties that would define an improved water reclamation system are:

1. Zero-g operating capability achieved without moving parts.
2. Capability of Recovering one hundred percent (100%) of all available water.
3. Provision for removing solids from the waste stream that presents a minimum health hazard to the crew and has a minimum adverse effect on the operational efficiency of the system.
4. Recovery economy better than 38 watt-hrs per pound for average recovery of up to ninety seven percent (97%) of all available water.
5. Elimination of all expendable items.
6. A system weight of less than 20 pounds per pound of water recovered per hour.
7. Elimination of all entrainment carryover.
8. A system that by means of its simplicity and good engineering and design is basically reliable and capable of many months of trouble-free service.

1.2 **System Objectives**

The objectives of the program conducted under this contract were to design an improved system for the recovery of water based upon the concept of a membrane vapor diffusion still and to construct a laboratory model of this system. The system constructed was to have the following specific performance capabilities:

1. To reclaim at least one pound of water each hour under conditions of one-or zero-g acceleration.
2. To achieve this rate of reclamation through evaporating and condensing at temperatures within the range of 32°F to 240°F, and with a minimum of moving parts.

1.2

(Continued)

3. To be capable of continuous operation and to include the necessary controls to sustain operation.
4. That the basic design of the system have as its design objective continuous operation for at least six (6) months without significant maintenance.
5. That the expendable items be minimized.
6. That the method of urine feed into the system and withdrawal of recovered water be such that they require a minimum expenditure of energy.
7. That the design parameters of the system be optimized.
8. That heat exchangers be at least 90% efficient.
9. That the ability of the system to meet the above specific objectives be demonstrated by means of a 100 hour continuous test.

2.0 SYSTEM CONCEPT

The concept selected for development is called a "membrane vapor diffusion still". This concept employs evaporation and condensation to produce potable water from chemically pretreated urine. A semi-permeable membrane is used as a low impedance, high entrainment efficiency, zero-g phase separator. The evaporation and condensation is accomplished in a passive manner with high thermal efficiency by means of vapor pressure diffusion across a low pressure air gap. The water vapor is condensed and separated from the air by means of porous metal condensing water separator. The urine from which the water has been extracted is continuously recirculated behind the membrane until the solids concentration becomes prohibitively high, at which point it is replaced with a fresh charge, the expended brine being discharged to the spacecraft solid waste disposal system.

2.1 Diffusion Still Module

A schematic cross section of a diffusion still module is shown in Figure 1. Starting at the center and moving out in either direction, the module consists of the following elements:

1. coolant fluid flow passage
2. product water collection passage
3. porous metal plate condensing surface
4. vapor diffusion gap
5. semi-permeable membrane
6. urine flow passage
7. heating fluid flow passage

Pretreated urine is continuously circulated through the urine flow passages behind the membrane. The flow of heating fluid supplies the required heat to the urine to raise it to the desired operating temperature and to permit evaporation of its water content at an acceptable rate.

The water contained in the urine is absorbed by the semi-permeable membrane at its liquid interface and evaporated from its opposite surface. The evaporated water vapor diffuses through the gas in the vapor diffusion gap and condenses on the surface of the porous plate. The pressure of the gas in the diffusion gap is maintained slightly above ambient to force the condensate through the porous plate and into the product water collection line. In the absence of excess water at the porous

2.1 (Continued)

plate surface, the existing liquid sets up capillary forces that resist the leakage of the gas from the diffusion gap. The concentrated urine is recirculated until the maximum practical quantity of the available water has been recovered.

The driving potential for this process is the water vapor partial pressure gradient which is established by the temperature difference between the heated urine and the condenser surface. The resistance to flow consists of the permeabilities of the membrane and diffusion gap.

The water vapor partial pressure in the brine is a function of its solids concentration as well as its temperature. There is also an effect of the solids distribution in the brine at the surface of the membrane.

2.2 System Description

A complete system for the reclamation of potable water from human urine is shown schematically in Figure 2.

The modules are supplied with the recirculating urine flow from the batch tank (4) by the urine feed pump (6). The batch tank and the vapor diffusion gap of the modules are held at slight positive pressures by means of regulated gas supply (P). The urine circulating pump draws the urine through the module and discharges it back into the batch tank, maintaining the flowing urine in the module at a pressure slightly lower than the pressure in the air gap. This causes the membrane to be held in place against the tops of the channels in which the heated urine flows. The product water is discharged from the porous metal condenser plate and through a water conductivity cell (8) for a quality determination and then to a canister containing activated charcoal (10) for a final removal of trace contaminants and odor. The product is then stored in a tank (11) which is vented to cabin atmosphere. In the event that the water conductivity cell indicates a poor product, the conductivity monitor will actuate the diverter valve (9) and the product water will be returned to the urine supply system for reprocessing.

Upon subsequent product improvement, automatic deactivation of the diverter valve will return the system to normal flow.

When the desired amount of water has been extracted from a batch of urine, the concentrated waste is discharged into the spacecraft solid waste disposal system by means of the 3 way valve (5). Fresh charges of urine are supplied from the collection and pre-treatment tank (3) and are fed into the batch tank by means of this same 3 way valve. The batch and collection tanks are sized to hold one day's waste collection. The collection and pretreatment tank is kept at a pressure (Pt) slightly above that of the batch tank and is fed urine from the waste collection system intermittently. The collection

2.2 (Continued)

tank initially has a premeasured quantity of pretreatment chemical injected into it by means of the chemical injector (2) from the chemical storage tank (1). Thus, when the collection tank is filled with urine, the proper ratio of chemical and urine results, and this new batch is ready for transfer to the batch tank for processing.

The system constructed under this contract consists of items (4), (5), (6), (7), (8), and (10). Figures 3-6 are photographs of the system and a vapor diffusion still module.

The batch tank (4) is a zero-g stainless steel accumulator with a chemically resistant bellofram. The exposed metal surfaces are treated with a fluorocarbon coating for improved chemical resistance. This item was designed at HSD for previous applications.

The brine pump (6) is a commercial laboratory item. The pumping element consists of a combination of bellows and check valve of one piece molded acid-resistant plastic construction. An oscillator coil and spring compress and expand the bellows 60 times per second, the check valve permitting flow in one direction only.

The diffusion still assembly (7) consists of five modules, containing two brine flow paths each, to provide the desired processing rate of 1 lb/hr. The modules are supplied with heating fluid, cooling fluid, and gas pressurization in parallel and from common headers. The brine flow through the modules may be connected either in series, in parallel or in a combination of both.

The product water is collected in a common header and passed through the conductivity sensor which monitors the quality of the product.

The conductivity sensor (8) is a commercially available item containing a cell with platinum electrodes and a bridge output circuit calibrated against a laboratory standard at HSD.

The water charcoal filter (10) is a component that has been designed at HSD for an air evaporation water reclamation system. Its purpose is to remove trace impurities and thereby improve pH, odor and color of the product water.

3.0 SYSTEM DESIGN

Prior to the undertaking of this effort, laboratory tests were made to ascertain the feasibility of the concept (Ref. 1). A porous plate sublimating heat exchanger which had been developed for another HSD program was used as the condenser-water separator. Cellophane membranes were attached to the condenser with their surfaces separated by open pore urethane foam sheets to form the vapor diffusion gap. The whole assembly was immersed in a tank of heated urine and a slight suction was applied to the condenser outlet.

These tests indicated a processing capability of .250 lb water/hr-ft² membrane for an overall vapor pressure difference of 2.68 psi. The quality of the product water (250 mho/cm), was not as high as had been expected and this was attributed to the formation of water bridges along the surfaces of the urethane foam these resulted in an osmotic transference of undesirable urine constituents into the product.

The pretreatment chemicals and process used and the post-treatment water charcoal are the result of previous experiences at HSD with air evaporation water reclamation systems.

3.1 Condenser-Water Separator Design

The porous plate condenser-water separator used in this system is a modification of a sublimating heat exchanger designed and built by HSD and available as off-the-shelf hardware.

The active surfaces of these sublimators consist of porous sintered metal plates. The parameters used to describe the performance of these plates for the present application are gas pressure bubble point with water and the liquid permeability of the plate to water.

The bubble point is the pressure necessary to force gas through the water filled pores and is determined by the surface tension of the water, the pore size opening and the type of gas and metal used. The Skau-Ruska equation relates these parameters in the following ideal manner:

$$P_{\text{bubble point}} = \frac{2 \cos \theta}{r}$$

where σ is the liquid-vapor surface tension at a particular temperature condition, θ is the contact angle of the fluid in contact with both the porous material and the gas, and r is the radius of the pore opening.

The rate of liquid flow, r , through a porous plate is proportional to its cross sectional area, A , and the pressure difference across the plate, ΔP , and inversely proportional to the thickness, t , of the plate. For the laminar flow regime it is given by Darcy's Equation:

3.1 (Continued)

$$R = \frac{KA \Delta P}{t}$$

where the proportionality constant, K, is a function of the porosity of the porous plate, the tortuosity of flow paths, the effective passage diameters, and the physical properties of the flowing liquid.

The contact angle, θ , is a function of the type of material and, in particular, the condition of the surface of the material. The size, shape and distribution of the pores are a function of the raw material powder particle size, the degree of compacting and the sintering schedule. The only feasible method of determining the suitability of a particular material is to test it and measure its bubble point and permeability performance with water.

Figure 7 shows the actual permeability to water of a typical commercially available sintered porous metal material as a function of the bubble point measured by the manufacturer (Ref. 2).

The design requirements for the water separator condenser were the ability to pass a flow of water of .25 lb/hr-ft² at the design pressure differential of 1 psi, and to have a bubble point of twice that pressure. As can be seen from Figure 7, for all reasonable plate pore sizes, the permeability is far in excess of that required and the choice of plate can be based upon bubble point requirements. On this basis, a plate of 5 micron pore size or smaller would be sufficient.

Samples of the porous nickel used in the manufacture of the sublimating heat exchanger were obtained and tested to establish their actual permeability and bubble point with water. Results of these tests are shown in Figure 7, and although the measured permeability was substantially lower than expected, the resultant values are still 25-250 times that which the design requires.

The thermal design of the sublimating heat exchanger provides a total thermal conductance from the condensing surface of the porous plate to the cooling water of:

$$U_{\text{condenser}} = 300 \frac{\text{BTU}}{\text{Hr-ft}^2\text{-}^\circ\text{F}}$$

For the design value of heat flux per unit of surface area of 446 Btu/hr-ft² the resultant temperature difference from coolant to condensing surface would be 1.5 °F.

The structural design of the sublimating heat exchanger allows a pressure of 60 psig in the coolant passage. A flow rate of water of 280 lbs/minute is estimated to have 50 psig

3.1 (Continued)

pressure drop in the coolant passage. For this flow and the design heat flux of 439 Btu/hr the cooling fluid temperature rise would be 1.6 °F.

The materials used in the all-brazed construction of the sublimating heat exchanger are nickel and stainless steel, providing a high degree of resistance to corrosion in the humid environment of the condenser.

The overall dimensions of the sublimator are 6.5" x 10.0" x 0.3" with a porous plate and water cavity on each side, and a common coolant passage. To adapt this item to the function of a condenser-water separator for the diffusion still only flanges and headering had to be added. The flanges are "picture frames" of stainless steel which, when brazed to either side of the porous plate module, provide a mounting and sealing surface for the membrane and a controlled gap between the membrane and the condensing surface. The flange also contains the gas pressurization port to provide the diffusion gap pressure. The coolant and water delivery ports use the flanges as closures in the headering arrangement.

The resultant design has an effective surface area of 57 in² per porous plate. Figure 8 is a photograph of a completed condenser-water separator.

3.2 Membrane Selection

The selection of a suitable membrane for use in the diffusion still application was based upon the results of an HSD-sponsored test program discussed in a later section of this report.

A number of membranes, which from published information appeared to have good possibilities for this application, were experimentally evaluated. Of these, only cellophane showed sufficient permeability to be attractive.

The ideal membrane should pass water with negligible pressure drop, have a high rejection capability for everything other than water, and be chemically resistant to the service condition as well as mechanically durable.

Cellophane is a semi-permeable membrane of a hydrophilic nature. Its capability to transmit water when a driving potential is applied is characterized by a permeability constant stated for a given thickness as:

$$K_m = \frac{\text{rate of water flow}}{\text{unit of area, unit of pressure difference}}$$

This permeability constant is a function of both the diffusivity of water vapor and the solubility of water in the membrane. The value of the permeability constant is a

3.2 (Continued)

function of the manner in which it is measured. Published information (Ref. 3, 4, 5, and 6) on the water permeability of cellophane for vapor-membrane-vapor systems and for liquid-membrane-liquid systems are as much as three orders of magnitude apart, and one published report (Ref. 7) on a liquid-membrane-vapor system indicates water permeabilities two orders of magnitude higher than either of the above referenced systems.

The results of the HSD experimental evaluation based upon a liquid-membrane-vapor system are shown in Figure 23. The water permeability, with pure water, of a number of cellophane membranes of different thickness is shown as a function of liquid temperature. Also shown is data for water permeability from urine as determined by HSD and by Warner, Brown and Glass (Ref. 7). Data for an all-liquid system by Lonsdale, Merton et al, (Ref. 5) is indicated and for an all-vapor system by Doty, Aiken and Mark (Ref. 3).

On the basis of this data, a design permeability of $.32 \text{ lb/hr-ft}^2\text{-psi}$ at 140°F was chosen. At the desired rate of $.288 \text{ lb/hr-ft}^2$ at 140°F , the vapor-pressure drop across the membrane is 0.90 psi.

The manufacturer's specification (Ref. 4) describing the chemical and physical properties of cellophane lists its resistance to acids as poor. The pretreated urine has a pH of about 3.4 and is considered highly corrosive. Samples of membranes soaked in pretreated urine of various solids concentrations showed no discoloration or sign of deterioration after 250 hours of 140°F exposure. When these membranes were mounted in a test fixture and stressed while subjected to the above exposure, severe discoloration occurred in the areas of maximum stress.

Cellophane is a surprisingly durable and resilient material, provided care is taken to avoid contact with sharp edges or points. While wet, the membrane may be deformed to more than three times its original dimensions before rupture occurs.

If sufficient force to deform the membrane 20-30% is applied and then removed, the original dimensions are very nearly regained after a period of time, provided the membrane has been kept moist.

3.3 Urine/Brine Management

The manner in which the urine or brine to be processed is supplied to the diffusion still has significant effects upon the successful operation of the still.

The driving potential for the process is the water vapor partial pressure difference across the membrane and diffusion gap. The vapor partial pressure for the water content of the urine brine is a function of the fraction of solids present and the temperature as shown in Figure 9 (Reference 8).

3.3 (Continued)

The brine, in passing through the diffusion cell system, will have its fraction of solids increased because of the removal of a portion of its water content. The ratio of the solids fraction at the inlet to that at the outlet is a function of the rate of brine circulation and the rate of water removal as shown in Figure 10. In order to maximize performance, the average solids concentration should be kept low. For the desired single module rate of 0.2 lb/hr, Figure 10 shows that for brine flow rates of 10 lb/hr or higher the inlet-to-outlet ratio of solids fractions will be .98 or greater. The desired recovery of 95% of the available water would result in a bulk brine of .50 solids fraction. If this is assumed to be the solids concentration entering the module at 10 lb/hr for the desired processing rate of 0.2 lb/hr, the exit solids fraction would be .51.

The penalty associated with the brine circulation rate is the pump power necessary to circulate the desired brine flows against the brine system pressure drop. Figure 11 shows the pressure drop of the brine flow path through one half of a diffusion cell as a function of brine flow rate and brine fraction of solids. Figure 12 shows the performance characteristic of the laboratory pump chosen to circulate the brine. For 95% recovery, the solids fraction would be .5, and with the brine passages for the two halves of each module connected in series and all five cells connected in parallel, an equilibrium brine flow of 48 lb/hr per module would be required. The total pump requirements would be 240 lb/hr at a head of 0.3 psi. With an efficiency of 10% for a flight weight pump, the power required to pump brine would be 2.0 Btu/hr or .59 watts.

If all 10 brine passages are connected in series, this pump would develop 2.5 psi head and would circulate 75 lb/hr of brine. The required pump power for this would be 5.1 Btu/hr or 1.5 watts. Besides requiring more power, this arrangement would impose a more severe mechanical strain upon the membranes.

The batch tank for the brine has been sized to contain a 20 hour charge based upon a processing rate of 1 lb/hr. This charge, through continuous recirculation, will have 95% of its water content removed and its volume reduced by a factor of about 13 to 1. This concentrated brine would then be discharged to the spacecraft solid waste system. The charging and discharging operation occurring once each 24 hour period is accomplished by means of a manual 3 way valve.

Figure 13 shows the brine Reynolds Number as a function of the brine flow rate and the brine solids fraction. The brine flow is laminar for the conditions expected in the diffusion still. In order to assure uniform distribution of solids locally in the brine flow, a high degree of turbulence must be induced by the evaporator brine flow path design.

3.4

Diffusion Gap

The diffusion gap is the space between the membrane and the condenser-water separator. Its function is to allow the water extracted from the brine to pass in the vapor phase from the membrane to the condenser, thus avoiding the transfer of undesirable urine constituents. It also serves as a thermal barrier between the hot urine and the condenser.

The transfer of water vapor by diffusion requires a concentration gradient. For gases and vapors, the concentration gradient is equivalent to a partial pressure gradient. In the case of the diffusion still, the gradient is maintained by the temperature difference between the brine and the condenser. The vapor pressure of water in the brine at the brine temperature and the vapor pressure of water at the condensing temperature sets up a gradient which causes water to flow from the brine through the membrane and, as vapor, across the diffusion gap to the condenser. The membrane offers impedance in this flow path as do the gas molecules in the diffusion gap.

The membrane impedance is defined by the membrane permeability constant K_m , which relates the rate of flow through the membrane to the pressure drop across the membrane in the equation

$$R = K_m A_e (P_1 - P_2);$$

where A_e is the effective membrane area, P_1 is the water vapor partial pressure in the brine and P_2 is the water vapor partial pressure on the air side of the membrane. The membrane permeability constant design value is .32 lb/hr-ft²-psi for cellophane with urine, as discussed in the sections on membrane selection and membrane testing.

The one dimensional diffusion equation used to describe the flow of water vapor through a non-condensable gas is written:

$$R = \frac{D_{12} A P (P_2 - P_3) M}{R_o T P_{B_m} (Y_2 - Y_1)}$$

where R is the diffusion rate (lb/hr)

D_{12} is the diffusivity of water vapor through the gas (ft²/hr)

A is the diffusional cross sectional area (ft²)

P is the total pressure (lb/ft²)

P_2 is the partial pressure of water at the vapor side of the membrane (lb/ft²)

P_3 is the partial pressure of water at the condenser (lb/ft²)

M is the molecular weight of water (lb/mole)

R_o is the universal gas constant (lb-ft/mole-°R)

T is the average gap temperature (°R)

P_{B_m} is the log mean partial pressure of the diffusion medium (lb/ft²)

$(Y_2 - Y_1)$ is the gap width (ft)

3.4 (Continued)

The value for the diffusion coefficient of water vapor through air of $0.99 \text{ ft}^2/\text{hr}$ at 560°R and atmospheric pressure was used for all of the calculations in this design.

The simultaneous solution of the membrane equation and the diffusion gap equation determines the processing rate for a given system.

In the present concept, a maximum usable membrane area of 50 in^2 per half cell and a brine temperature of 140°F were chosen as design values. The membrane area is approximately 12% less than the condensing area due to an allowance for the supporting of the membrane. The 140°F brine temperature is the maximum permissible (Ref. 9) consistent with the reclamation of good product water. At higher temperatures, urine decomposes, releasing gaseous products which diffuse along with the water vapor. The gas in the diffusion gap, for this design, is nitrogen.

In order to maximise the processing rate, the diffusion gap should be as small as possible and the condensing temperature as low as possible. Both of these parameters will also effect the heat loss from evaporator to condenser and therefore must be the subject of an optimization study as discussed later.

Figure 14 shows the solution of the membrane and diffusion gap equations for a half cell as a function of brine solids fractions and diffusion gap width. The brine temperature used was 140°F and the condenser temperature was 60°F . As was discussed previously under brine management, the expected exit solids fraction for 95% recovery is .51 and the required half cell process rate is .1 lb/hr. From Figure 14, a diffusion gap of .01 ft meets the design performance requirements.

The performance of the vapor diffusion still could be increased by the use of a gas other than nitrogen. The diffusivity of water vapor through helium, for example, would be approximately 2.5 times that through nitrogen. Helium, having a much higher thermal conductivity (5X) would have a much higher thermal loss due to diffusion gap thermal conduction, as discussed under Thermal Design. The final choice of gas or mixture of gases would depend upon the results of a future optimization study.

3.5 Evaporator Design

The evaporator to be used in the system has to provide a means of supporting and sealing the membrane material, maintaining an effective flow of urine beneath the membrane, transferring heat from the heating fluid to the flowing urine and supplying sufficient structural rigidity to maintain a diffusion gap of constant spacing.

To minimize fabrication problems, it was decided to make the evaporator from a single material, thus eliminating possible areas of corrosion at bonds between dissimilar materials. Plastic materials were eliminated due to their poor thermal

3.5 (Continued)

conductivities. Tests of various metals, described in Section 4.2 below, indicated AISI 347 stainless steel as the best choice consistent with relative ease of fabrication.

To support the membrane, provide maximum usage of membrane surface area, and provide a flow path of sufficient length for good heating and proper cross-section for good mixing of the urine, a serpentine flow path separated by narrow membrane-supporting ribs was evolved (Figure 15). The resultant arrangement provided approximately 170 inches of flow path and 50 square inches of useful membrane area in the less than 57 square inches of diffusion gap area provided within the condenser flanges. This flow path, as well as the heating fluid chamber on the opposite side of the evaporator (Figure 16) and a central mounting flange were machined from a single piece of material. Fin stock to create turbulence in the heating fluid and a cover for the heating fluid chamber were welded in place. A polyvinyl chloride thermal gasket provided a sealing surface and thermal insulation between the evaporator and condenser flanges. Mounting bolts, threaded directly into the condenser flange, were thermally insulated from the evaporator by fiber washers.

The heating fluid flow path has been estimated to have a 1.0 psi pressure drop with the perforated fin stock in the passage and a water flow of 200 lb/hr. For this flow and the expected heat transferred, the inlet to outlet water temperature difference will be 1.1°F. If all of the heat required for the evaporation of the desired rate of water and the conductive and radiative losses from the membrane is transferred from the evaporator base to the membrane by conduction through the brine and the membrane support ribs, the difference in temperature between membrane and evaporator base would be approximately 3°F. The high degree of tortuosity provided in the brine path with 28-180° bends not only provides good mixing to insure uniformly distributed local solids but will reduce this temperature differential to 1-2°F.

3.6 Thermal Design

The energy required for this process consists of the heat needed to raise the temperature of the incoming brine from the ambient (assumed 75°F) to the operating temperature of 140°F, the heat needed to evaporate 95% of the available water contained in this brine and the heat needed to make up for the thermal losses.

The thermal losses consist of internal conduction and radiation as well as external heat transfer. The external heat transfer has not been considered since small quantities of insulation can make this negligible. The internal losses are a function of the diffusion still design and consist of conduction through the gasket separating the condenser and evaporator flanges and the bolts connecting these two items; and conduction and radiation from the evaporator surface to the condenser surface through the diffusion gap.

3.6 (Continued)

The energy required can be represented by the following equation:

$$\text{Energy Required} = Q_i = Q_{H+} + Q_E + Q_{L1} + Q_{L2} + Q_{L3} + Q_{L4} + Q_{L5}$$

where Q_{H+} is the heat required to heat the brine from room temperature to the system operating temperature

Q_E is the heat required to evaporate the required water

Q_{L1} is the heat loss by conduction through the evaporator-condenser thermal spacer

Q_{L2} is the heat loss by conduction through the bolts which fasten the evaporator to the condenser

Q_{L3} is the heat conducted through the diffusion gap separating the membrane and condenser surfaces

Q_{L4} is the heat radiating from the membrane surface to the condensing surface

Q_{L5} is the external heat loss.

For the required process rate of .2 lb/hr per module and a 95% recovery of available water, a brine weight flow of .222 lb/hr is required. For a brine temperature of 140°F and a condenser temperature of 60°F, the required heats are calculated as follows:

Q_{H+} The urine entering the system is approximately 95% water by weight. The specific heat of the solids is negligible and therefore a specific heat at constant pressure of 0.95 was used. To raise the urine from an ambient of 75°F to the desired 140°F will require 13.7 Btu/hr per module.

Q_E The evaporation of the required water rate at 140°F requires latent heat of evaporation of 202.8 Btu/hr per module.

Q_{L1} The purpose of the thermal spacer is to act both as a gasket and a thermal conduction impedance. It is made of PVC with a total cross sectional area of 20.6 in² and a thickness of .22 inches. The heat loss through the spacer is 60.4 Btu/hr per module.

Q_{L2} There are 44 bolts which clamp the flanges of the evaporator and condenser on either side of the thermal spacer. These bolts have a thermal gasket under their heads to reduce their heat conduction loss. Heat transmission from the evaporator flange to the bolt has been considered to be by conduction through the gasket and through the gap between the bolt shank and the hole through the flange. The total loss through the bolts is 25.8 Btu/hr per module.

3.6 (Continued)

Q_{L3} The close spacing of the membrane and the condenser surface offers a low impedance to the conduction of heat through the gas filling the gap. This conduction loss is a function of the thermal conductivity of the gas and water vapor in the gap as well as the total exposed surface areas. For a gap spacing of .01 ft and a surface area of 114 in² and a bulk thermal conductivity of .0154 Btu/hr-ft²-°F/ft, the total conduction loss through the diffusion gap is 97.6 Btu/hr per module.

Q_{L4} The radiation loss from the membrane surface to the condenser surface through the diffusion gap is based upon the Boltzman Law. The membrane is assumed to have an emissivity of .9 and the condenser .5 with a surface area of 114 in². The total radiation loss through the diffusion gap is 36.2 Btu/hr per module.

Q_{L5} The external heat loss to the atmosphere can be reduced to a negligible quantity by the use of insulation. A polyurethane foam type insulation 1" thick can reduce the heat loss from a module to approximately 6.5 Btu/hr per module or 1.5% of the heat supplied.

The total design point energy, Q_i , required is:

$$Q_i = 13.7 + 202.8 + 60.4 + 25.8 + 97.6 + 36.2 + 6.5$$

$$= 443 \text{ Btu/hr per module}$$

The minimum heat required to affect the process is:

$$Q_{H+} + Q_E = 216.5 \text{ Btu/hr per module}$$

The energy rejected by the condenser consists of the heat required to cool the water vapor from 140°F to the condensing temperature and the latent heat of condensation plus the sum of the internal heat losses.

This can be represented by the following equation:

$$\text{Energy Rejected} = Q_o = Q_{H-} + Q_C + Q_{L1} + Q_{L2} + Q_{L3} + Q_{L4} + Q_{L6}$$

where Q_{H-} is the heat required to cool .2 lb/hr of vapor from saturation at 140°F to saturation at the condenser temperature of 60°F and is 6.8 Btu/hr per module.

3.6 (Continued)

Q_C is the heat of condensation at the condensing temperature of 60°F, or .2 lb/hr of water and is 212 Btu/hr per module.

Q_{L6} is the heat transferred into the condenser from the environment. This is considered negligible, due to the small temperature difference (15°F) and the almost negligible exposed area (25 in²).

Q_{L1-4} are as defined before - 220 Btu/hr per module.

The total design point energy rejected in the condenser is:

$$\begin{aligned} Q_0 &= 6.8 + 212 + 220 \\ &= 438.8 \text{ Btu/hr per module} \end{aligned}$$

The choice of condenser temperature will affect both the process rate and the thermal losses. Lower temperatures will result in an improvement in process rate but will increase the thermal loss.

The largest thermal loss item is the heat conduction through the diffusion gap. The process rate can be increased by reduction of the gap spacing but this also will increase the thermal losses.

A system optimization as a function of condenser temperature and diffusion gap width is discussed in section 5.0.

3.7 Acceleration

The system is composed entirely of gravity-independent components and a minimum of moving parts. Besides the flowing brine, the only moving parts are the brine circulation pump and the "rolling bellofram" within the zero-g tankage. The membrane, although flexible, is retained in place by the maintenance of the diffusion gap pressure in excess of the liquid pressure. Since the brine pressure drop is of the order of .5 psi or less with a vapor gap pressure of 1.5 psi, there is a net pressure of 1.0-1.5 psi on all portions of the membrane. The presence of one g along the vertical axis actually complicates the situation by requiring the head due to liquid height to be accounted for. For the .02 g lateral acceleration, the resultant force of the liquid against the membrane results in a pressure increase of 2×10^{-4} psi. For liquids contained in the variable volume "bellofram tanks", the maximum pressure due to the .02 g acceleration would be less than .02 psi.

4.0 TEST PROGRAM AND DISCUSSION OF RESULTS

4.1 Membrane Testing

The membrane in the vapor diffusion still serves as a zero-g liquid-vapor phase separator. In the normal evaporation situation, the rate of evaporation from the surface of a liquid is controlled by the vapor pressure of the liquid at its surface. This vapor pressure is a function of the temperature and composition of the liquid surface. In a membrane system, the membrane imposes a penalty not present in the case of normal evaporation. The membrane is a physical and chemical interface that offers additional resistance to the flow of fluid and therefore requires additional driving potential.

The details of the physics of the membrane are not completely understood, and opinions expressed in the literature vary diversely. It may be that the membrane is porous with capillary paths (Ref. 10) through which the fluid and some of the dissolved solids flow. The evaporation is then from a water layer on the surface of the membrane which contains impurities which lower the vapor pressure of the water. The water may also flow through the membrane by means of a process such as hydrogen bonding (Ref. 11) and the evaporation may be from a surface consisting of a weak chemical combination between membrane molecules and water molecules, again with a lowering of the vapor pressure. The nature of the process by which the fluids enter the membrane is equally unclear. It may be that the phenomenon involves chemi- or physi-sorption with a change in available driving potential. Any one or a combination of similar processes may be involved.

In the past, membrane performance has been described in terms of a coefficient of performance called a permeability constant, K_m , stated for a membrane of a specified thickness as:

$$K_m = \frac{\text{rate of permeation}}{\text{unit of area, pressure difference}}$$

A cellophane membrane was chosen for the initial diffusion still testing at HSD, the decision being based upon the work of Warner, Brown and Glass (Ref. 7). This study evaluated 28 potential membrane materials, of which cellophane had the highest permeability rates. Their data was based upon vapor being transmitted through the membrane in the same direction as the total pressure gradient, the discharge being to a vacuum. They found a water permeation rate for .0013 inch thick cellophane of 0.5 lb/hr-ft²-psi from urine at a temperature of 117-118°F. A comparison with data taken by Lonsdale and Merton et al (Ref. 5); 4x10⁻³ lbs/hr-ft²-psi for a .0009 inch thick membrane, in a reverse osmosis liquid-membrane-liquid system and with data from Doty, Aiken and Mark (Ref. 3); 4x10⁻⁵ lbs/hr-ft²-psi for a .0014 inch thick membrane, and DuPont (Ref. 4); 1x10⁻² lbs/hr-ft²-psi for a .0014 inch thick membrane, in a vapor-membrane-vapor system shows a substantially higher

4.1 (Continued)

permeation constant for the Warner, Brown and Glass data. Although the above referenced data was obtained at room temperature with water and the Warner, Brown and Glass data at 118°F with urine, there would still appear to be a substantially higher permeation rate for the liquid-membrane-vapor system.

The present application uses the membrane in a liquid-membrane-vapor environment with a minimum total pressure difference across the membrane in a direction opposite to that of the water vapor flow.

An evaluation of cellophane over a wide range of parameters with both water and urine was conducted at HSD (Ref. 12) as well as evaluations of other candidate materials. The program was company sponsored and is reported here because of its direct application to the design of the diffusion still system.

The test program was set up to determine the effects of operating temperature and membrane thickness upon membrane performance for cellophane and other membranes.

The basic method for determination of the necessary data utilizes air evaporation from the vapor side of the membrane to obtain membrane-limited performance. Nitrogen of low dew point is forced across the surface of the membrane in sufficient quantities that the rate of process fluid transmission is limited only by the membrane, that is, the rate of permeation becomes constant for increasing gas flow.

A membrane test fixture was fabricated from 347 stainless steel. This fixture (Figures 17 and 18) consists of an upper chamber which contains the fluid, (either water or urine), separated from the lower chamber by the sample of membrane. The membrane is clamped between two flanges, one a highly polished surface and the other containing an "O" ring. The lower chamber contains an inlet gas plenum, with a shower head arrangement to allow the incoming gas to impinge uniformly upon the membrane surface area.

The gas and water vapor leave the lower chamber through the central exhaust port. Provisions for measuring the pressure in the gas space under the membrane as well as the temperature of the incoming and exiting gas were included. The temperature of the liquid in close proximity to the liquid-membrane interface was also measured. A motorized stirrer was used to keep the liquid well mixed when urine was used.

The test fixture was installed in a constant temperature oven and the incoming gas supplied through an electric heater, (Figures 19 and 20). The gas supplied was nitrogen from a liquid tank farm. Its flowrate was measured with a venturi meter, and the rate of flow of the fluid being processed was determined with a pipette connected to the liquid plenum above the membrane, and a stop watch. The membrane was supported by means of a slight pressure differential (1.5" H₂O) maintained constant in a direction opposite to the direction of permeation.

4.1 (Continued)

A thermal balance was maintained by pre-heating the incoming gas and adjusting its temperature to insure that the heat required to evaporate at the resultant process rate was supplied entirely by the gas. This insured that the thermistor in the liquid also represented the membrane temperature.

As a control, the membranes were evaluated first with liquid water and then with urine on the feed side of the membrane.

Since the permeability of cellophane was substantially higher than that of any of the other membranes evaluated, it was chosen as the material for use in the diffusion still. Only the data for cellophane is presented here.

Permeation rates were measured for both pretreated urine and water as a function of nitrogen gas flow for a number of temperatures. This data has a great deal of scatter and Figures 21, and 22 represent typical smoothed curves. These curves all achieved a membrane-limited condition. Figure 21 shows membrane performance with water at room temperature and at 140°F. for two thicknesses of cellophane membrane. Figure 22 shows membrane performance with water and with urine at both room temperature and 140°F. This data was reduced to yield a membrane permeability constant. Figure 23 shows permeability constant as a function of temperature for a number of membrane thicknesses. Curves 1, 2, 3, 5, and 6 were obtained with the previously described test apparatus. Curves 4 and 7 were obtained with two different apparatus both discharging into a vacuum. Also noted on this figure for comparison are representative values for liquid-membrane-liquid systems and vapor-membrane-vapor systems.

4.2 Materials Selection

The hardware used to investigate, test and evaluate this new concept had to be durable and trouble-free under the wide range of abuses to which new hardware is normally subjected as well as resistant to the severe corrosion, exposure to hot, concentrated, chemically-pretreated urine may cause in a great many materials.

An experimental exposure to simulated service conditions was conducted for a number of metallic materials in an HSD sponsored program.

The candidate structural materials were corrosion tested in urine which had been pretreated with 4 ml of chemical per liter of urine. The pretreatment chemical consisted of 44% sulphuric acid, 11% chromium trioxide and 45% water by weight. Specimens, in plate form, cleaned and polished to 16 rms finish, were submerged in individual pyrex beakers of the urine and held at a temperature of 140°F by means of a water bath. Additional quantities of pretreated urine were added twice a day to replenish evaporated water, thereby increasing the concentration of the solids at a rate similar to that which would be experienced in actual service conditions.

4.2 (Continued)

The acidity of the urine brine, initially measured at a pH of 3.4 was maintained acidic and varied from 3.4 to 4.1.

Two individual corrosion investigations were performed. The first or preliminary test of 10 different alloys was to eliminate candidate materials which showed excessive general corrosion and pitting corrosion tendencies. The alloys tested are shown in Table 1 along with reasons for rejection or for acceptance into the more quantitative second test.

The second test was performed in a manner similar to the preliminary investigation, but with more exact control of chemistry, additives, and acidity of the solution. The test duration was over 300 hours, and was equivalent to a 90% water recovery. Table 2 includes corrosion rate data and metallurgical examination data on all samples. Corrosion rate data is reported in milligrams per square decimeter per day. Carpenter 20, an austenitic stainless steel not included in the preliminary test was also tested in this group because of its past performance in oxidizing acids.

All six materials tested in this section showed some protective qualities for the test duration. No evidence of pitting corrosion or heavy scaling was noted in any of the samples tested. Corrosion rates were moderate to negligible and all under 1 mil per year. Of all the alloys tested, only titanium revealed any discoloration. All other specimens evidenced some scale formation but displayed adequate resistant qualities.

All alloys tested are classified as weldable. Hastelloy C and titanium offer the greatest difficulty in machining, with Incoloy 825, Inconel 600 and AISI 347 stainless steel next most difficult. Titanium appears to give the greatest advantage from a weight consideration.

The AISI 347 stainless steel was chosen for this application due to its good corrosion resistance, better machinability and availability.

The use of fabrication techniques such as brazing were ruled out and welding was minimized to avoid the severe galvanic corrosion of dissimilar metals which can occur in the presence of pretreated urine brine which is a very strong electrolyte.

4.3 Component Testing4.3.1 Evaporators

Upon receipt of the evaporator sections of the diffusion still module from the shop, some simple tests were performed to develop confidence in the design. The purpose of these tests was to observe the membrane under the design pressure differential with circulating brine at the design temperature and high salt concentrations.

4.3.1 (Continued)

A Cellophane membrane was mounted on the evaporator using the thermal-spacer and a spare flange from a condenser. A brine pump was connected to the evaporator and pretreated urine circulated at approximately 23 lb/hr from an open storage tank in a manner which maintained approximately a 1.5 psi positive pressure differential across the membrane in the direction from the air to the liquid. The evaporator was maintained at 140°F and a ventilation hood was used to remove the water vapor as it evaporated from the membrane surface. The rate of evaporation was measured by the decrease in volume of the storage tank. The brine volume was replenished periodically with additional pretreated urine. Periodically, samples of the brine were taken and a solids fraction determined.

Initial tests were conducted using .9 mil and 1.6 mil membranes with a urine pretreatment level of 4 ml/liter of HSD pretreatment chemical (9). The tests with the thinner membrane were terminated within a 100 hour exposure due to membrane failure. Based upon the membrane evaluation program, the performance of the 1.6 mil membrane was considered sufficient for the system requirements and all of the subsequent testing was with the thicker membrane.

A number of problem areas were observed during this testing. Any pockets formed between the membrane and the evaporator structure outside the circulating brine passages, if filled with brine, would result eventually in a membrane failure. The evaporation from the pocket through the membrane and the lack of subsequent mixing with the bulk brine developed concentrations in the pockets which were extremely high. The most significant occurrence of these pockets was in the assembly clearance space between the thermal spacer-gasket and the evaporator brine channel outer periphery. Occasionally, these pockets would occur on the top surface of the membrane support ribs. Evidently, small imperfections in the surface, once wet with brine, would siphon additional fluid into the void and develop an increased solids concentration. The effect on the membrane of these high brine concentrations was found to be caused by the urine chemical pretreatment. A test of 250 hours duration was run without any urine pretreatment. At the end of this time, the membrane showed no signs of failing. An alternate HSD pretreatment chemical was evaluated but the membrane was so severely attacked by it that the test had to be terminated in 25 hours.

The solution of this problem was approached in three ways:

- a. The void areas where the brine concentrations occurred were filled with an RTV silicone rubber compound.
- b. A mylar shield was overlaid on the membrane in the peripheral areas where these pockets had occurred to reduce the evaporation through the membrane in these areas.
- c. The amount of pretreatment chemical used was reduced to the stoichiometric requirement of 2.5 ml per liter. The 4 ml level had been established in the air evaporation program at HSD (9) and was in excess of the stoichiometric

4.3.1 (Continued)

requirement due to operating characteristics of that system.

Tests were run with the silicone rubber filled voids and the reduced pretreatment level for a continuous 250 hour period with no membrane failure. There were, however, indications of solids build up in the peripheral areas and it was at this time that the mylar shield was added. This configuration was not tested until the pre-acceptance test which is described later in this report.

4.3.2 Condensers and Modules

Each condenser module as it was received from the shop was tested for its minimum bubble point with water and its water permeability. Although the modules showed considerable variation in their permeability, all had more than adequate capability to pass the required .2 lbs/hr under a 1.0 psi differential. The measured values are shown in Figure 24 and are of the same order of magnitude as the material samples tested during the design phase.

The first complete module received from the shop was assembled and tested with water to determine its processing rate. A rate of .264 lbs/hr was obtained with a 140°F evaporator temperature and 60°F condenser temperature. Small amounts of water were observed in the diffusion gap pressurization lines indicative of either a leaking membrane or a poorly performing condenser. Vegetable dye food coloring was added to the feed water to trace the source of the water in the gap. A very slight discoloration of the product water and the water in the gap pressurization line indicated the probability of the formation of a water bridge between the condenser and the membrane, since a leak would have been indicated by a more severe coloration.

Upon module disassembly, the condenser was set up above a hot water bath to observe the condensation pattern on the porous plate. A small pump provided a vacuum on the delivery port, supplying the driving force for the transfer of the condensed water through the porous plate. Small, clearly defined areas where condensation but no transfer of water occurred were observed, with the condensed water hanging dropwise with the plate in a horizontal position. With the plate in a vertical position, these droplets ran to the bottom of the plate, collecting on and eventually overflowing the flange. Enclosed in a module, this water would form a bridge between the membrane and the porous plate providing a path for the diffusion of dissolved solids through the membrane by osmosis.

Similar testing of the other condensers showed some areas on both sides of each condenser which exhibited this failing. Further, it was found that these areas were highly permeable to the flow of gas and could not be sealed against the flow of gas by being pre-wetted with water.

4.3.2 (Continued)

This behavior suggested the local contamination of the plate by oil or grease or some other hydrophobic substance.

A large variety of cleaning techniques were used to try to clean these areas. Liquid, vapor and ultrasonic cleaning as well as acid and caustic cleaning baths were tried, without success. The plates could be sealed against the leakage of gas by first wetting the plates with alcohol and then replacing the alcohol with water by soaking them in a bath of water. These plates still exhibited the local surface hydrophobicity.

It was decided to try to find a coating for the condenser surface that would be hydrophilic in nature, with good lateral wicking capabilities in order to transfer the condensed water from inactive sites to locations at which it could be absorbed. Various materials were tried on sample pieces of porous plate and evaluated to determine their wettability, wicking properties and degree of blockage to the plate porosity. The only material which appeared to solve the problem was a fluoro-chemical surfactant rewetting agent, HSD-PS67 (Ref. 14), which is applied in a toluence solution and then baked to remove the solvent. This material forms a mono-molecular layer on the surface to which it is applied and enhances the wettability of that surface. Previous tests of this material (Ref. 15) had shown a bubble point reduction in porous plates of about 50-60%, but this did not present a problem in this case because of the excess capacity of the plates in this respect.

The method of coating the condensers consisted of a 250°F bake for 10 hours to completely dry the porous plate, a freon ultrasonic cleaning for 1 hour and then the application of the PS 67. A liberal amount was poured onto the plate until no further absorption took place. Dry nitrogen was forced through the plate and the excess liquid was sponged off until no trace liquid was in evidence. The plates were then baked in a 250°F oven for 10 hours to remove any residual solvent from the porous plates.

If the porous plate condenser should malfunction by developing the hydrophobic surface areas or by becoming locally blocked, the condensed moisture would collect until the droplet size was sufficient in a one-g field to run down the plate, collecting at the lower edge. This liquid would form a water bridge across the flange and allow the passage of dissolved solids into the porous plate condenser. These solids would then plug the porous plate and prevent the removal of the water bridge. In order to prevent such contamination and possible destruction of the porous plate condensers due to one-g malfunction, the lower edge of the mylar shield was extended 1/2 inch into the diffusion gap to form an enclosure in which excess water would collect between the porous plate and the shield instead of forming bridges to the membrane surface.

4.3.2 (Continued)

A second area of difficulty which was investigated was an indication that the permeability of the porous plate tended to decrease with use. In order to investigate this phenomenon the plates were completely immersed in a tank of water and a suction head applied to the water delivery line. The rate of water passing through the porous plates as a function of time was measured for a number of applied suction pressures, and it was found that the rate did decrease with continued exposure. This was at first thought to be the effect of gas which was dissolved in the water, nucleating within the body of the porous material and effectively blocking the flow path through the pores. Tests with water which has been deaerated in a vacuum chamber still showed the decreasing performance with time. All of the porous plate permeability data previously obtained had been at pressure differentials and water flows orders of magnitude higher than those required for the component. A test was run for 250 hours at approximately twice the required process rate and resulted in permeability values which fluctuated both up and down but remained in excess of the required performance. The changes in permeability observed could have been influenced, if not caused, by bacteria colonies growing in the water and depositing on or in the porous plate, causing blockage. However, this would not be a problem in the diffusion still since the water vapor comes from a sterilized liquid and is filtered through the membrane, transported as a vapor and condensed on the surface of the porous plate. The resulting product, therefore, should be bacteria and spore free.

4.4 System Testing4.4.1 Pre-Acceptance Test

A single module was assembled and tested in order to establish that the modifications to the evaporator and condenser had satisfactorily eliminated the known problems. The system urine batch tank and brine circulating pump were connected to the module and the brine passages of the two halves connected in series. The heating requirements for the evaporator were supplied by a small pump circulating the water from a thermostatically controlled hot water bath, while the condenser was cooled by tap water at approximately 60°F. The hot water bath temperature was adjusted to maintain the urine outlet temperature at 140°F. The heating fluid, cooling fluid, and urine inlet and outlet temperatures were monitored throughout the test. Hourly readings were taken of the fluid temperatures, heating and cooling fluid flow rates, and the product water volume, pH, and conductivity during normal working hours. The test was run unattended at all other times. Figure 25 shows the test set-up. Curves of processing rate, product water conductivity, and product water pH plotted as a function of elapsed time are shown in Figures 26, 27, and 28. The product water was collected without charcoal post treatment.

This test was run with an initial urine volume of about 6.6 liters which was replenished after 42 hours with an additional 4.5 liters. The concentration ratio at the conclusion of the test at 85 hours was 8.21 or about 33% solids. The average

4.4.1 (Continued)

processing rate of 0.258 pounds per hour for the 85 hour duration of the test was well in excess of the 0.2 pounds per hour per module required of the final five module system.

The product water collected from 20-36 hours elapsed time was manually filtered through a laboratory charcoal bed until the pH was 7.0. The product water collected from 43-60 hours elapsed time was sent, untreated, along with the charcoal filtered sample to an independent laboratory for analysis. A tabulation of the results of the analyses of these two samples is presented in Table 3.

Product water quality was acceptable throughout the test; its conductivity ranging from 47 to 186 micromhos per centimeter and its pH ranging from 5.36 to 6.28. Slight urine odor, coloration, and turbidity were eliminated in the sample filtered through charcoal. Impurities in the analyzed samples were well within the limits imposed by the United States Public Health Service (Ref, 16) for drinking water with the exception of manganese, discussed further in Section 4.5.5.

4.4.2 Acceptance Test

In order to demonstrate the Improved Water Reclamation System's capability to meet the design objectives listed in Section 1.2, a 100 hour continuous test was performed. Since the complete system was not fully assembled at the time of the acceptance test, it was decided to test a single module and scale the acceptance process rate to that of a 1/5 system, .2 lbs/hr.

A single module was assembled and tested in the same manner as in the pre-acceptance test with the exception that the system charcoal canister was connected to the product water delivery line and the test set up was placed in an enclosure to limit external heat transfer. A glass flowrater was installed in the brine circulating line to measure the brine flow rate. All measurements were made as outlined in the pre-acceptance test, except that the test was monitored continuously.

To provide a baseline for later comparison of water quality, the system was charged initially with tap water and run for about 18 hours before draining and recharging with pretreated urine to begin the 100 hour test. The water collected during this baseline run was refrigerated and later sent to an independent chemical laboratory for analysis.

To begin the 100 hour test, 8.1 liters of pretreated urine were introduced into the urine supply tank. 200 milliliters of residual tap water in the module, left from the baseline testing, mixed with the urine reducing the solids concentration by a small fraction, (from about 4.75 to 4.64 percent) but having no other effect. This dilution was negated and the original solids concentration restored in less than two hours of processing.

4.4.2 (Continued)

An additional 7.3 liters of pretreated urine were added to the tank after 41.5 hours. This total of 15.4 liters introduced contained 33.51 pounds of available water and 1.65 pounds of solids. 28.43 pounds of water were removed from the system during the duration of the test for an average processing rate of 0.284 pounds per hour, well in excess of the required rate. A curve of processing rate plotted versus elapsed time is presented in Figure 29.

The brine circulation rate, plotted in Figure 30 as a function of elapsed time, began to decrease gradually at about the 20 hour point in the test. Although the cause of this was not apparent, the system performance did not seem to be affected; and the test was allowed to continue until at the 82 hour point, the brine flow had almost stopped and the process rate began to decline. On the theory that the precipitation of solids in the brine was causing a degradation in the performance of the brine circulating pump, a spare pump was installed with an upstream settling chamber to prevent the solids from entering the new pump. This did not correct the problem but the test was allowed to continue until the 100 hour point. The low brine flow rates and the decline of the processing rate disrupted the thermal equilibrium of the system, and it became necessary to increase the hot water temperature to maintain the brine outlet temperature at 140°F. This resulted in a period of excessive processing rate followed by a decline which continued until, at the 100 hour point, the process rate had dropped to just below the .2 lbs/hr required rate.

Curves of product water pH and conductivity versus elapsed time, indicative of the instantaneous water quality, are presented in Figures 31 and 32. Results of the laboratory analyses of product water collected during hours 41-69 and 82-100, and the pre-test baseline product water are presented in Table 3 with a tabulation for comparison of the impurity concentration limits imposed by the United States Public Health Service for drinking water.

The abrupt breaks in both the pH and conductivity curves at 82 hours are coincidental with the removal of the water charcoal canister from the system, effected to obtain an unfiltered sample of the product water for analysis.

At the completion of 100 hours, the test was interrupted to investigate the cause of the low brine circulation rate. The problem was found to be a blocked outlet fitting from the batch tank and therefore the module inlet line was connected to the tank drain fitting as an alternate. At this time, the settling chamber was removed from the brine flow path, careful note being made as to the quantity of fluid removed with it and stored for later analysis. Upon resumption of the test, the brine flow rate returned to its initial value. In 16 hours of additional testing, the process rate recovered and was initially higher than expected, due to excessive urine temperatures, but returned to the expected rate after thermal control has been re-established. The rate then decreased until the test was terminated.

4.4.2 (Continued)

The system was then drained of brine and refilled with tap water and run with a brine outlet temperature of 166°F and a condenser temperature of 66°F. A process rate of .625 lbs/hr was recorded. The brine temperature was then reduced to 100°F and a process rate of .116 lbs/hr recorded, and then further reduced to 74°F with a condenser temperature of 68°F yielding a process rate of .09 lbs/hr.

Upon disassembly of the test set-up, it was found that a batch tank outlet fitting made of aluminum had been used instead of the specified stainless steel. This fitting, shown in Figure 33, had become completely plugged with the products of galvanic corrosion brought about by the dissimilar materials in contact with the strongly electrolytic brine.

The module disassembled at the conclusion of the test is shown in Figures 34 and 35. The membranes were in very good condition in comparison to the membrane tested in open air evaporators. There was no evidence of membrane deterioration in the troublesome areas previously described in Section 4.3.1.

4.5 Discussion4.5.1 Membranes

The data obtained from the membrane testing program for the membrane water permeation rate from urine to the vapor phase displayed considerable scatter and a great deal of judgement was used to draw the curves of permeation rate as a function of dry nitrogen flow (Figures 21 and 22). The permeation constant, K_m , calculated from this data and plotted as a function of temperature, (Figure 23) showed consistent correlation for both water and urine of a decreasing permeability with increasing temperature. The HSD data (Ref. 13) taken with an apparatus that discharged the water vapor to a vacuum also showed this decreasing characteristic, although these values appear low since the membrane thickness was intermediate between the ones tested during this program. Two points from data published by Warner, Brown and Glass (Ref. 7) of water permeation from urine through a membrane to vacuum also appear to be consistent with the data obtained for urine in the membrane test program.

The decrease in permeability with increasing temperature is contrary to what was expected on the basis of published information on permeability of materials to gases (Ref. 17). The permeability constant for gases can be shown to be proportional to both the solubility and the diffusion constant of the gas in the material. Cellophane is a hydrophilic material and exhibits a characteristic sorption and desorption isotherm for water vapor. Although the only published data found to date (Ref. 18 and 19) are at room temperatures, the general theory of adsorption indicates a decreasing solubility with increasing temperature. The room temperature adsorption of liquid water by cellophane has been reported (Ref. 4, 5 and 18) to vary between 49-70% of the weight of the dry material, and if the data published by McBain and Stuewer

4.5.1 (Continued)

(Ref. 20) on the swelling of cellophane at room temperature (25°C) and at 100°C can be considered indicative of the amount of water adsorbed in the membrane, there is a 46% reduction at 100°C over that at 25°C. The high degree of permeability to water exhibited by cellophane is undoubtedly dependent on its extremely high sorption of water and the decrease of this sorption at increased temperature should be expected to reduce the permeation rate.

The test results from the pre-acceptance test and the acceptance test were used to calculate the membrane permeability constant, K_m . The two tests used different condensing water separator modules and there was a difference of over 19% between them in the average thickness of their machined flanges which set the diffusion gap dimension. The resultant values of K_m of .28 for the pre-acceptance test and .30 for the acceptance test are in good agreement with the chosen design value of .32.

4.5.2 Condensers

The use of the rewetting surfactant HSD-PS67 on the porous plate condensing water separator proved an exceptionally satisfactory solution to the problem of local hydrophobic surface areas. It not only eliminated this problem but improved the performance of the satisfactory areas by increasing their water wicking rate.

The problems of bacteria and gas embolism were not encountered in the module testing performed under this contract but may develop on test exposures of longer duration. The surfactant used does have fungicidal properties but it is not germicidal. It may however, be possible to find a suitable compound for this application which would be germicidal if this should prove necessary.

The mono-molecular film which this material deposits has a very low solubility in water, of the order of a few parts per million. There will be a definite life time to the effectiveness of the film as a function of the quantity of water processed. However, other applications of this material at Hamilton Standard have shown life times of more than 600 hours and 400 lbs total flow per square inch of area with no loss of performance.

4.5.3 Thermal Analysis

The test data from the pre-acceptance, acceptance and post-acceptance testing was analyzed to determine the actual internal losses. The total heat rejected in the condenser was calculated from the cooling water flow and temperature rise. This heat rejection consists of the heat required to cool and condense the processed water and the heat transferred due to the internal losses. The process water must be cooled from saturated vapor at the temperature of the evaporator to that of saturated liquid at the condenser temperature. The difference between the heat rejected in the condenser and the heat actually required for the process is the total of all the losses, internal and external into the condenser.

4.5.3 (Continued)

The external losses would be almost negligible due to the small temperature difference between the condenser and ambient temperatures (15°F) and the small exposed surface area (25 in²).

The total internal losses calculated from the pre-acceptance test were 205 Btu/hr averaged for a number of test points which were representative of thermal equilibrium and stability. The predicted losses for the test conditions were calculated on the basis outlined in the section on thermal design for the test conditions and are 210 Btu/hr. The test and predicted losses are within 2-1/2% of each other and considered to be within the accuracy of the test data.

The acceptance test data resulted in measured losses of 260 Btu/hr and predicted losses of 230 Btu/hr. The measured loss data is 13% higher than the predicted losses and may be due to a higher than expected external heat input into the condenser. The test module had been installed within an enclosure for the acceptance test, and the ambient temperatures within the enclosures in the vicinity of the module may have been as high as 130°F.

4.5.4 Brine Management

Analytical performance predictions showed the performance reduction to be expected as the brine solids fraction increased (Figure 14). Based upon the initial volume of urine, residual volume of tap water, and the amounts of brine added and removed from the system during the acceptance test, a curve was constructed showing brine solids-fraction as a function of elapsed time during the test (Figure 36).

This curve was plotted by calculating the weights of water and solids introduced to the system in the pretreated urine at the initial and 41.5 hour fills. A minimum solids concentration of 4.75 percent was assumed, (reference 21) since it had proved consistent through previous urine collections at Hamilton Standard. The water left in the module from the base line water run was included in the initial fill, reducing the starting solids concentration to 4.64 percent. A sample of the brine removed at 100 hours was dried and its solids content determined. The weights of the urine inputs, with the weights of process water collected, provided a running balance of both water and solids in the system.

The brine drained from the system at the conclusion of the test was found to contain precipitated solids. The calculated concentration at this point was 69.4% solids. Since precipitates are not formed in urine brine at concentrations below 60% solids (Reference 8), it was assumed that the curve was within the normal limits of experimental accuracy.

The processing rate measured during the acceptance test has been plotted in Figure 37 as a function of solids fraction as determined from Figure 36. The analytically determined

4.5.4 (Continued)

process rate has been drawn through the data and shows remarkably good agreement. Those data points which deviate significantly from the analytical curve are indicative of poor thermal control of the test, with the exception of the series of points between 20-25% solids which occurred at about the 90-100 hour point in the test. The brine flow at this point was very low, about 2-3 lbs/hr with a Reynolds Number of about 80. It would appear from this that at 20% solids, the brine solids boundary layer could be sufficiently controlled by a Reynolds Number of about 100. At the termination of the test, the brine Reynolds Number was about 600 and the solids fraction was .69 with the test data showing no significant deviation from the analytical prediction.

Although the parameters which control the brine boundary layer solids concentration have not been sufficiently analyzed, it would not be expected that the effect of such a boundary layer would be negligible at these low Reynolds Numbers. The high degree of turbulence normally produced in the brine by the 56 flow reversals in the brine flow path had as its purpose the minimization of the boundary layer effects and must therefore be considered to have been effective.

The exact value of the critical Reynolds Number to minimize the brine solids boundary layer and its variation as a function of brine bulk solids fraction has neither been analytically nor experimentally investigated in this program. The minimization of the pump work required and the weight penalty associated with this work would be the result of efforts in this area. It is therefore recommended that this work be done in the future.

4.5.5 Water Quality

To be of acceptable quality for continued consumption, the water produced by any reclamation system should contain no impurity which would cause objectionable odor, taste or color; nor any impurity in a concentration which might be hazardous to the health of the consumer. The United States Public Health Service has established, on the basis of lifetime consumption, limits for the concentration levels of various chemical impurities to be tolerated in public drinking water supplies, as well as tolerable threshold intensities of odor, color and turbidity. A tabulation of these quantities for those impurities that may be expected to appear in water reclaimed from urine is presented in Table 3.

Also presented in Table 3 are the results of the analysis of two samples of water reclaimed from urine by the Improved Water Reclamation System during the 100 hour acceptance test, one sample processed from tap water just prior to the inception of the test and the two samples reclaimed from urine during pre-acceptance testing.

Each of the product water samples analyzed was below the USPHS limit for color and burbidity. While odor detection is not a part of the analysis procedure, random

4.5.4 (Continued)

observers all described the odor of the sample not filtered through charcoal as being that of faint urine or ammonia. No odor was detectable in the charcoal filtered sample.

The chemical impurities present in each sample were below the USPHS limits for all impurities except manganese, which was at or above the limit in every sample. The concentration of manganese in the two pre-acceptance test samples, collected during successive overnight runs, was 0.13 parts per million for the first charcoal filtered sample and 0.19 ppm for the second unfiltered sample. Successive samples taken during the acceptance test showed 0.1 ppm for the base line water run, 0.078 ppm for the charcoal filtered 41-69 hour sample and 0.05 ppm for the unfiltered 82-100 hour sample. Since all but the last of these concentrations is above the normal maximum manganese concentration in urine of 0.066 ppm (ref. 21), at least a part of the manganese in the product water must be from a source other than the urine. A review of the materials the product water contacts after passing through the condenser's porous plate revealed that both of the materials in the water collection chamber in the condenser, "A" nickel fin stock and MIL-5-6721 stainless steel sheet stock, contain manganese in 0.25 and 2.0% concentrations respectively. Calculations show that the average concentration level in the product water of 0.11 milligrams per liter could be achieved from this source alone if each liter of water took into solution just 0.1% of the manganese available in a 0.0002 inch depth of each of the manganese bearing metals. This does not take into account the additional manganese bearing metals. This does not take into account the additional manganese available from the stainless steel tubing and fittings in the processed water lines or the nickel porous plate. The constantly decreasing concentration of manganese, reduced to the USPHS limit in the last sample analyzed, indicates that the action of the product water in taking manganese into solution is the most probable cause of contamination and that further samples would have the concentration reduced to acceptable levels.

The analyses of the acceptance test product water do not include a determination of urea nitrogen. The analyses of the three samples delivered to the Newlands Laboratory on June 2 showed urea nitrogen concentrations of 100 parts per million for the base line water run sample and 900 parts for million for each of the urine run samples. Since these values were completely inconsistent with past experience, including the pre-acceptance test, where urea nitrogen concentrations of zero were the norm, it was decided to have the urea nitrogen determination repeated for verification.

Second samples of each of the acceptance test product waters originally analyzed, drawn from the balance of the volume collected during the test (which had been stored under refrigeration in the collection bottles from the time of collection) were delivered to the Newlands Laboratory on June 26 for rereadings of color, pH, conductivity and urea nitrogen concentration. A marked increase in the intensity of the odor and color of the unfiltered urine run sample was noted at this time.

4.5.5 (Continued)

The repeated readings of color, pH and conductivity for the base line water run and the charcoal filtered urine run samples showed little or no change from the original readings. Urea nitrogen concentrations of 10 parts per million and zero, respectively, for these samples was determined.

The unfiltered urine run sample displayed changes in color from 6 to 540 units (a pale yellow color), pH from 6.9 to 6.1 and conductivity from 203 to 630 micromhos per million was determined. The sample sent to the Newlands Laboratory on June 2 was also stored under refrigeration, the volumes of this sample, too small to be used for the determination of any of the repeated readings, was observed on June 26 to have retained its original color.

Since the change in color occurred only in that portion of the unfiltered urine run sample stored for an extended period in the collection bottle, it is reasonable to assume that some contaminant was present in this bottle to cause the changes in pH and conductivity as well as the change in color. The evidently erroneous original urea nitrogen concentration does not provide a valid basis for comparison, though the low readings on the other samples indicate that the high urea nitrogen concentration of this sample is probably a result of the same contamination.

5.0

SYSTEM POTENTIAL

The test results on the membrane diffusion still indicate that a four module system would be capable of meeting the 1 lb/hr requirement. A preliminary design for a prototype package was performed in order to estimate the weight and thermal performance for comparison with competitive systems. Figure 38 shows four modules integrated into a single assembly. The condenser water separators are of the same design as those produced for this contract with the exception that the porous plate thickness and the water collection passage gap have been reduced to that of a similar Hamilton Standard design which is presently made in a smaller total surface area. All of the materials and methods of manufacture would remain the same. The stainless steel flanges on the condenser have been replaced with a plastic spacer which is held in place by clamping and which will reduce the internal conduction heat loss. The overall cross sectional area of the thermal spacers has been decreased to reduce the internal losses. The six interior evaporators are supplied heat in pairs from three modular stainless steel heating sections of a similar construction to the cooling fluid cores in the condensers. The two exterior evaporators are heated by two additional cores. The brine flow passage is fabricated from a sheet of plastic, the exterior rim of which is the thermal spacer and gasket for the membrane. The whole assembly is contained between two aluminum rectangular flanges connected with tie bolts which clamp the assembly in position. These tie bolts connect surfaces which are at the same thermal potential and therefore there is no bolt conduction loss. The brine passages as well as the heating, cooling and vapor gap pressure ports are externally manifolded in parallel. The estimated weight for this four module assembly including the manifold and clamping frames is 16.25 lbs.

The additional components whose weight is considered chargeable to the system by being uniquely a part of the system (Figure 2) are: the batch tank (4), brine management valve (5), brine pump (6), conductivity sensor and control (8), product water diverter valve (9), and product water charcoal canister (10). The chemical storage tank (1) and chemical injector (2) have also been included as chargeable weights although all systems most probably will have parts of a similar nature. The total system fixed weight is therefore estimated to be 29.63 lbs.

The performance for this four module system was calculated for four condenser temperatures for the original diffusion gap spacing of .01 ft and for a diffusion gap of .005 ft. This data is shown as a function of brine solids fraction in Figure 39. In order to determine the system processing rate, it was assumed that the system batch tank was charged with 24 lbs of pretreated urine and starting with 4.75% solids, the area under the curve was integrated with respect to time until the residual brine was at 50% solids. The water recovered, being 95% of that available or 21.62 lbs, was then divided by the time required to accomplish this brine concentration yielding an average processing rate for the batch. A system fixed weight per man was then calculated as a function of condenser temperature for the two diffusion gap spacings. These calculations were based upon the method outlined by Popma and Collins (Ref. 22) and employed the same thermal and electrical penalties used by these

5.0

(Continued)

authors. The thermal losses were calculated as described in the section on thermal design for the four module prototype design. The only electrical power required to operate this system is the power for the brine circulating pump and the power for the product water conductivity monitor and control. The pump power has been assessed on the basis of the power required to circulate the brine at a Reynolds Number of 1500 with a solids fraction of .50. This has been calculated to be 3 watts. The conductivity monitor and control has been assessed at .6 watts based upon similar equipment presently designed by Hamilton Standard. Figure 40 shows the results of this analysis. The effect of condensing temperature is quite small in the range of 40-80°F with a substantial decrease in performance above 80°F. The reduction in diffusion gap space increases the system process rate to a much greater extent than it increases the thermal conductance loss thereby reducing the system weight by approximately 11%. The .005' gap is considered to be within the present design capability for this type of hardware. For the .005' gap and a condensing temperature of 70°F, the system weight per man is 7.0 lb.

These calculations have been repeated for a diffusion gap of .005' for a 98% recovery of available water with a resultant system weight of 7.25 lbs/man.

The expendables required for this system consist of pretreatment chemical of .0040 lb chemical/lb water, post treatment charcoal of .00125 lb charcoal/lb water, and membranes .00058 lb membrane/lb water. The quantities of pretreatment chemical and post treatment charcoal are based upon requirements established at Hamilton Standard on air evaporation water reclamation systems. The requirement for membranes is based upon a life time of 100 hours. The membranes have been considered here as an expendable, although for a practical system the membranes must be capable of operating for the expected mission duration as a part of the system fixed weight. The membrane is such an intimate part of the system that the problems associated with replacing them in flight would make the system unattractive. For systems operating at 95% recovery the penalty in expendables must include .154 lbs stored water/day to make up for the unrecovered water. The ability to recover 98% reduces this penalty to .062 lbs water/day. Figure 41 shows the system launch weight as a function of mission duration.

The potential of further improvement in the system weight exists in the areas of choice of the gas or mixture of gases to maximize the diffusion process while minimizing the thermal conductance. Also the treatment of the surface of the condenser to reduce the thermal radiative loss.

Substantial improvement in system potential could be achieved by the use of improved urine pretreatment concepts which would allow an increase in evaporator temperature, and/or minimize the reduction in vapor pressure with increasing solids fraction.

5.0 (Continued)

There is an inherent performance increase for the system when applied to a spacecraft system operating at reduced total pressure. The diffusion gap pressure is approximately cabin ambient and therefore the diffusion gap permeability constant, K_g , will increase providing an increased processing rate with no attendant penalties. For a one third reduction in the gap pressure, it has been estimated that there will be a 23% reduction in the fixed weight of the system.

The two major areas which require further effort in order to realize the full potential of this system concept are:

1. The investigation of other existing membranes and the development of new membranes which will have improved chemical resistance and mechanical properties under the service conditions imposed by this concept. These membranes should have as high or higher permeability constant for water in a liquid/membrane/vapor system. The life time of the membrane should be sufficient to allow the membrane to be considered an integral part of the system, requiring no maintenance for the duration of the expected mission.
2. The brine management parameters should be investigated analytically and experimentally to determine the optimum method of minimizing the brine solids boundary layer at the membrane. Also improved techniques for determining the brine solids fraction and percent recovery of available water should be investigated and a standard procedure adopted.

TABLE 1

PRELIMINARY CORROSION INVESTIGATION IN PRETREATED URINE AT 140°F

1. **Martensitic Stainless Steel**
AISI 410 per AMS 5504 - Rejected for limited pitting corrosion after 151 hours
2. **Austenitic Stainless Steel**
AISI 347 per AMS 5512 - Acceptable with less than .1 mg/sq in change after 457 hours

Incoloy 825 - Huntington Alloys - Acceptable with less than 3 mg/sq in change after 264 hours
3. **Aluminum Alloys**
6061-T6 per AMS 4027 - Unacceptable because of excessive pitting and heavy corrosion after 150 hours. Anodized samples gave slightly longer life.
4. **Titanium Alloys**
Unalloyed Ti-70A per AMS 4901 - Acceptable with only light adherent corrosion film formation
5. **Cobalt Base Alloys**
Hastelloy B - Unacceptable because of general heavy corrosion pattern
Hastelloy C - Acceptable
6. **Nickel Base Alloys**
Nickel 200 - Unacceptable because of general medium corrosion and pitting corrosion
Monel 400 - Unacceptable because of heavy scaling and limited pitting
Inconel 600 - Acceptable with less than 2 mg/sq in change after 264 hours

TABLE 2CORROSION TEST DATA FOR EXPOSURE
TO PRETREATED URINE AT 140°F

<u>Specimen</u>	<u>Exposure Time in Hours</u>	<u>Corrosion Rate Milligrams per Square Decimeter per Day</u>	<u>Metallurgical Comments</u>
Hastelloy C	95	.0094	Surface etching visible
	170	.0027	Surface etching visible
	245	.0043	surface etching visible
	320	.0084	Staining and etching - no evidence of pitting
Titanium Ti-70A	95	.0010	Heavy etching of surface
	170	.0028	Light adherant scale
	245	.0039	Light adherant scale
	320	.0036	Light adherant scale
Incoloy 825	95	.0142	--
	170	.0022	Light surface etching
	245	.0015	Light surface etching
	320	.0012	Light surface etching
Inconel 600	95	.018	--
	170	--	Slight surface etching
	245	.0031	Slight surface etching
	320	.0012	Light scaling
347 Stainless Steel	95	.0010	--
	170	.0028	Light surface etching
	245	.0039	Light scaling
	320	.0021	Light scaling
Carpenter 20	95	.050	Light scaling
	170	--	Light scaling
	245	.0157	Light scaling
	320	.0150	Light scaling

TABLE 3

PRODUCT WATER ANALYSES

Item	Pre-Acceptance Test		Acceptance Test		USPHS Limits
	24-36 Hours	44-60 Hours	Tap Water, Charcoal	41-69 Hours Charcoal	
	Charcoal	No Charcoal			
Color	2	8	3	2	15
Turbidity	0	3	2	4	5
pH	7.0	5.4	7.3	6.5	10.5
Conductivity	96	67.7	183	79.9	N.S.
Alkalinity	39	30	70	36	N.S.
Hardness	18	1	64	32	N.S.
Oxygen Consumed	5.5	14	0.4	0.2	N.S.
Anionic Detergent	0.31	1.04	N.D.	N.D.	0.5
Solids, Volatile	17	25	18	20	N.S.
Solids, Fixed	54	25	92	50	N.S.
Solids, Total	71	50	110	70	500
Nitrogen, Ammonia	1.11	2	0.55	1.51	N.S.
Nitrogen, Albuminoid	0.084	0.22	0.06	0.15	N.S.
Nitrogen, Nitrite	0.002	0	0.005	0.024	N.S.
Nitrogen, Nitrate	0.10	0.05	0.106	0.016	10
Nitrogen, Urea	0	0	N.D.*	N.D.*	N.S.
Chloride	0.5	0.5	2	0.5	250
Chromium, Hexavalent	0	0	0.01	N.D.	0.05
Sulfate	0.8	0.2	0.4	1.2	250
Iron	0.038	0.22	0.02	0.01	0.3
Copper	0.02	0.03	0.03	0.056	1
Manganese	0.13	0.19	0.1	0.078	0.3
Phosphate	0.58	2.66	13.1	0.62	N.S.
Fluoride	0	0	0.1	N.D.	3.4

All quantities except color, turbidity, pH and conductivity are parts per million. Color, turbidity and pH are scale numbers and conductivity is expressed in micromhos per centimeter.

The letters N.S. in the USPHS Limits column indicate that no limits for these items have been specified.

The letters N.D. indicate that none of the particular items was deleted in the sample tested.

Analyses made by the Newlands Sanitary Laboratory, Hartford, Connecticut.

*See Section 4.5.5 on Water Quality.

TABLE 4

WEIGHT SUMMARY FOR PROTOTYPE

FIXED

4 Modules, manifold and frame	16.25 lbs
Batch tank	6.75
Brine pump	.50
Brine management valve	.43
Diverter valve	.33
Conductivity sensor	.25
Conductivity circuit	.10
Charcoal canister	.37
Chemical storage tank	2.50
Chemical injector	.75
Component integration	<u>1.40</u>
	29.63 lbs.

EXPENDABLES

Pretreatment chemicals	.0040	lb/lb water
Post-treatment charcoal	.00125	lb/lb water
Membranes	.00058	lb/lb water

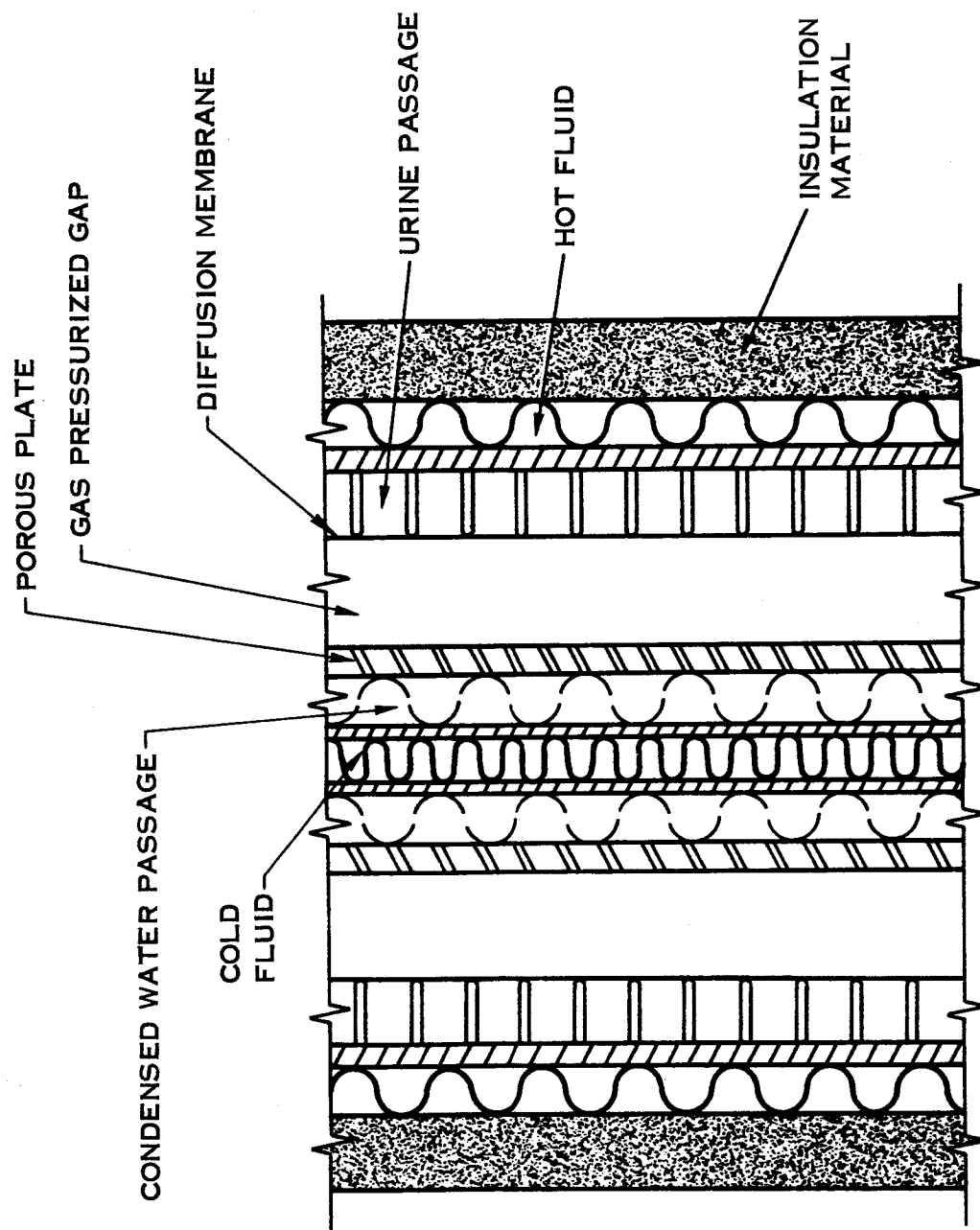
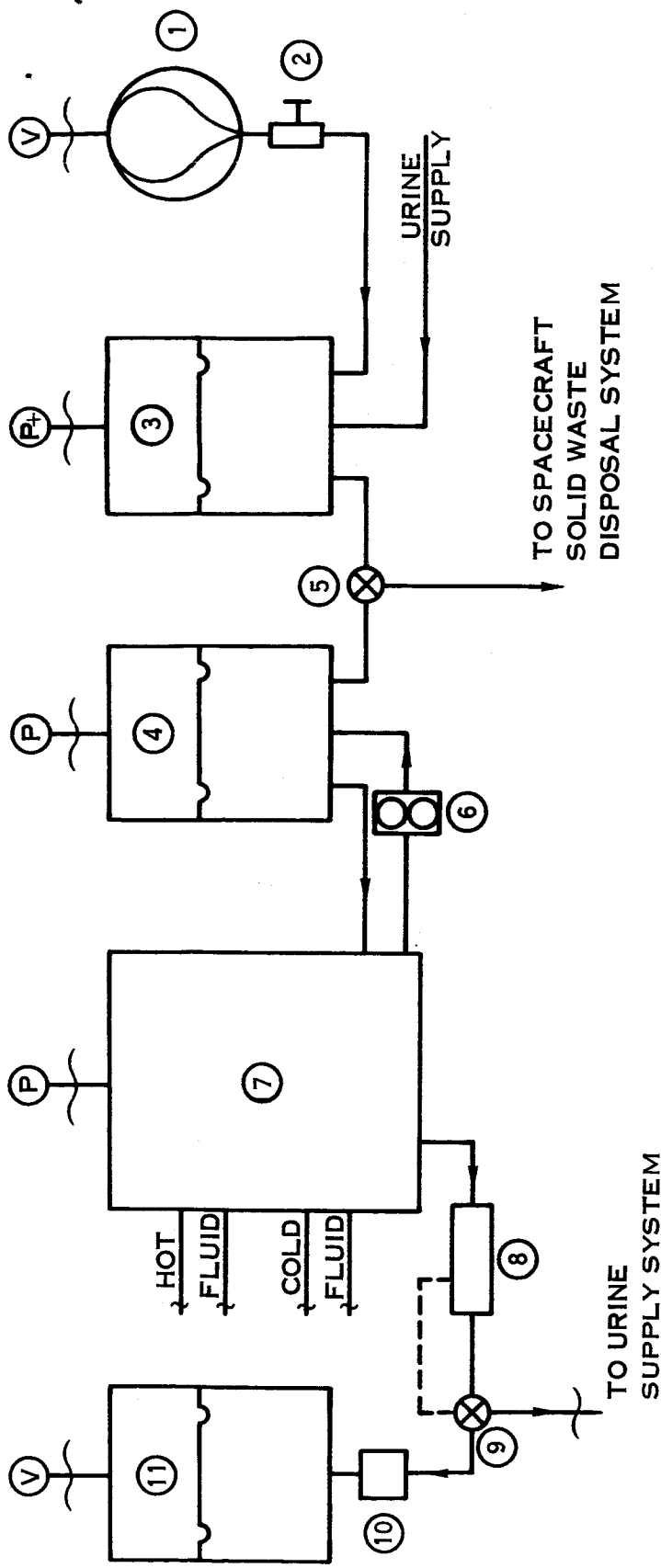


FIGURE 1. CROSS SECTION OF A DIFFUSION STILL MODULE - SCHEMATIC



1. CHEMICAL STORAGE TANK
 2. CHEMICAL INJECTOR
 3. URINE COLLECTION AND PRETREATMENT TANK
 4. SYSTEM BATCH TANK
 5. 3-WAY BRINE MANAGEMENT VALVE
 6. URINE CIRCULATION PUMP
 7. DIFFUSION STILL ASSEMBLY
 8. PRODUCT WATER CONDUCTIVITY SENSOR
 9. PRODUCT WATER DIVERTER VALVE
 10. PRODUCT WATER CHARCOAL FILTER
 11. PRODUCT WATER STORAGE TANK
- (V) TANK ULLAGE VENTED TO CABIN
 (P) TANK ULLAGE PRESSURIZED 1.5 PSIG ABOVE CABIN AMBIENT
 (P+) TANK ULLAGE PRESSURIZED IN EXCESS OF 1.5 PSIG ABOVE CABIN AMBIENT
 ~ SYSTEM INTERFACE

FIGURE 2. DIFFUSION STILL SYSTEM - SCHEMATIC

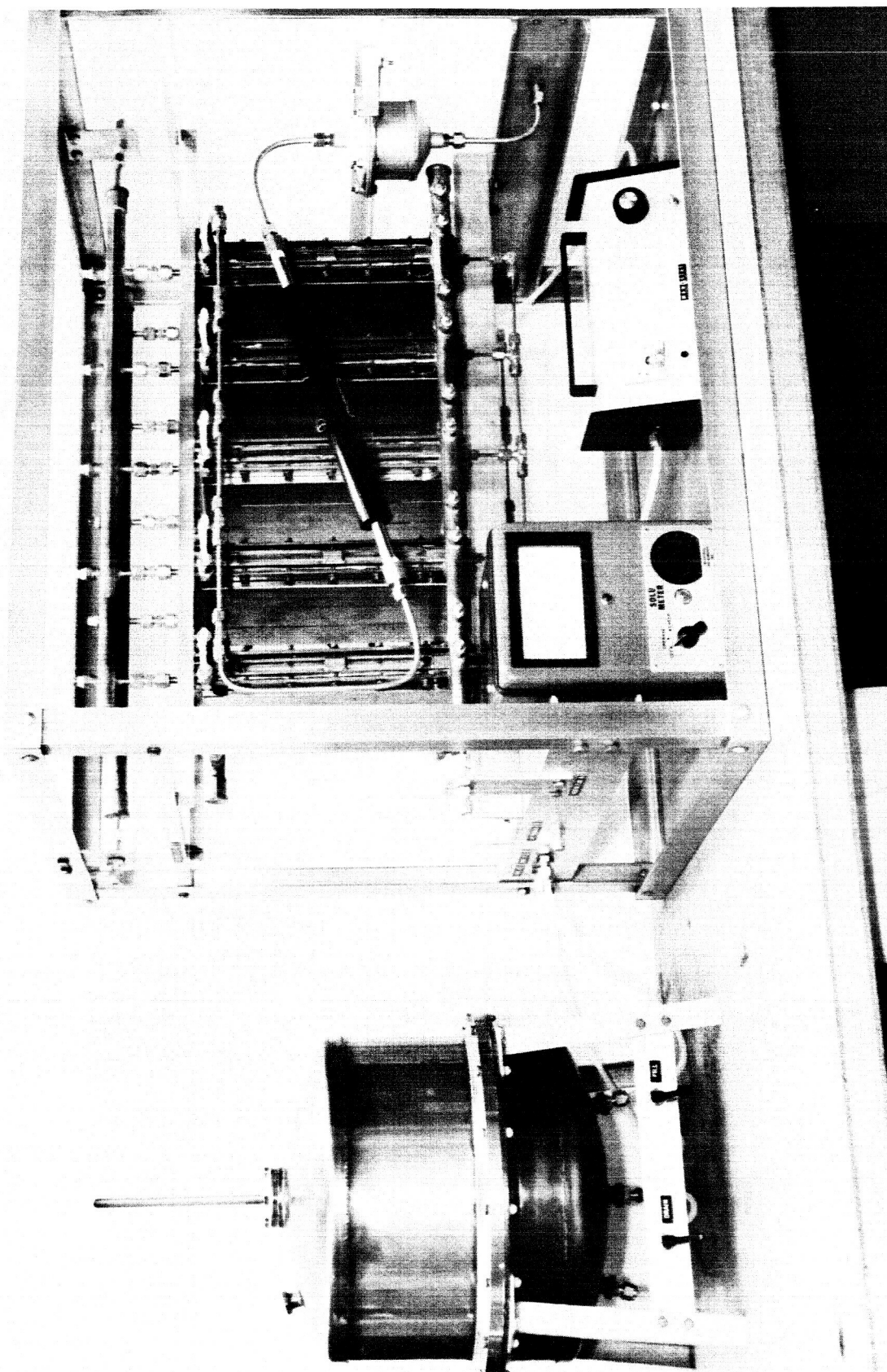


FIGURE 3. DIFFUSION STILL SYSTEM — FRONT VIEW

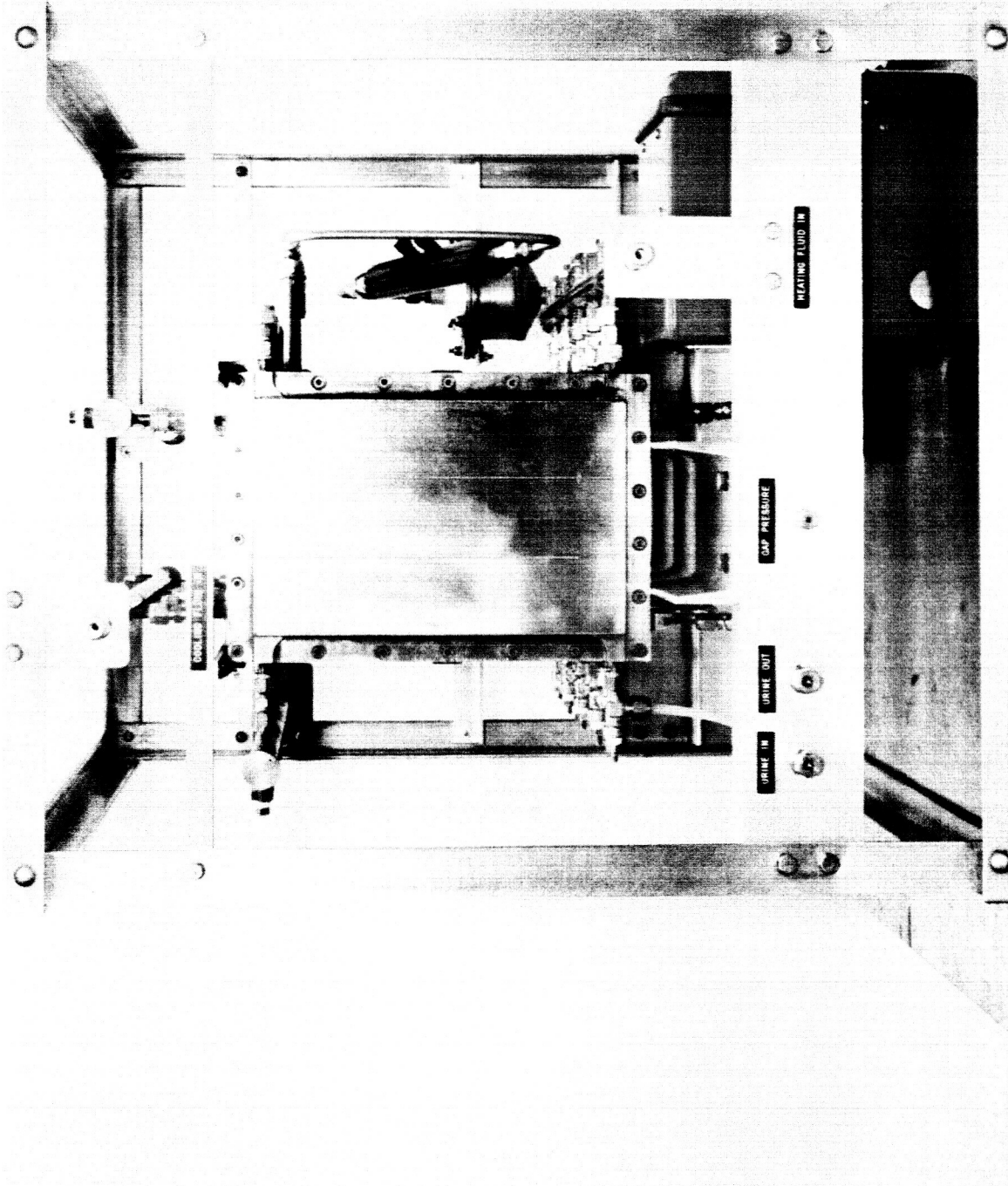


FIGURE 4. DIFFUSION STILL SYSTEM - SIDE VIEW

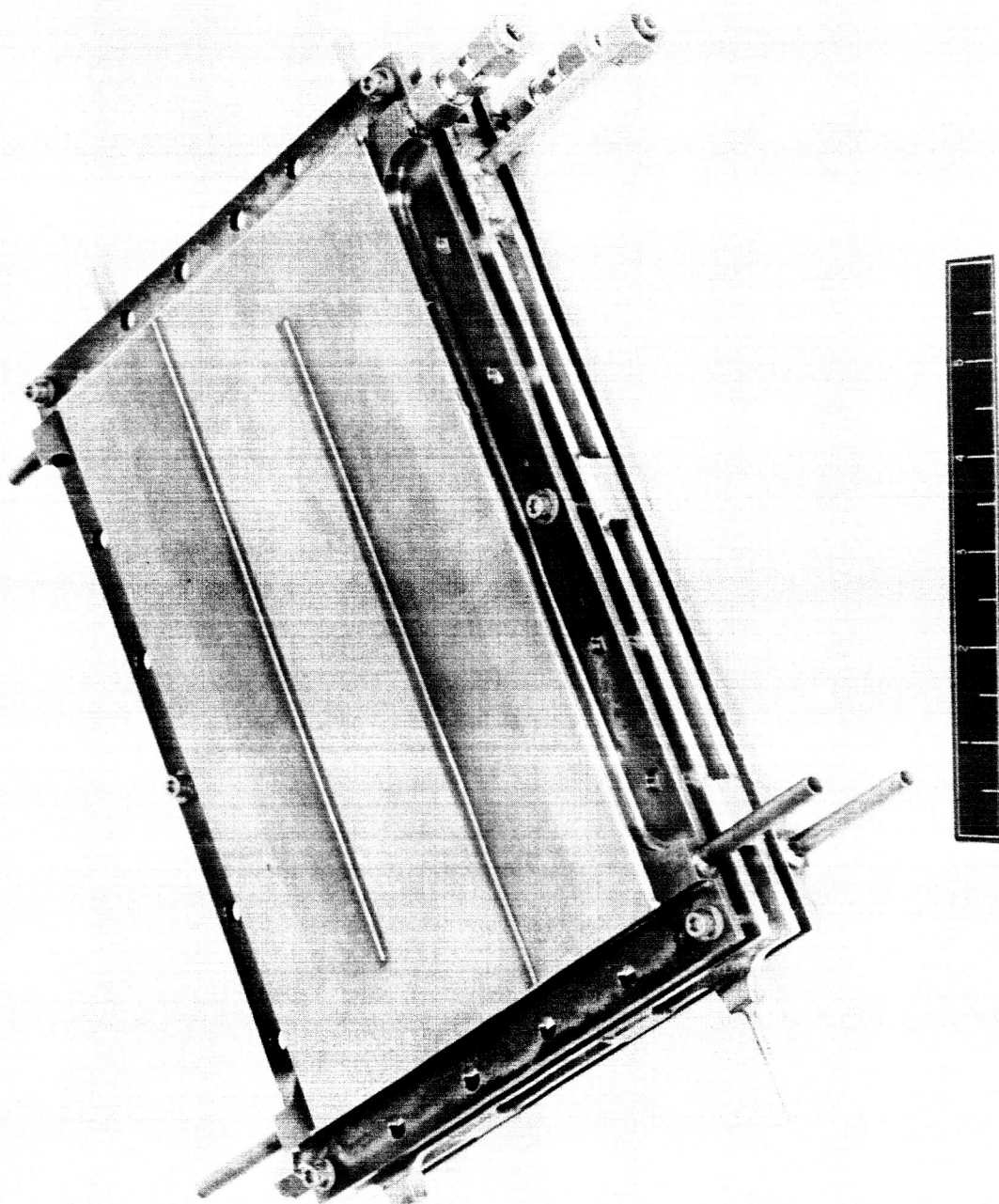


FIGURE 5. DIFFUSION STILL MODULE — ASSEMBLED

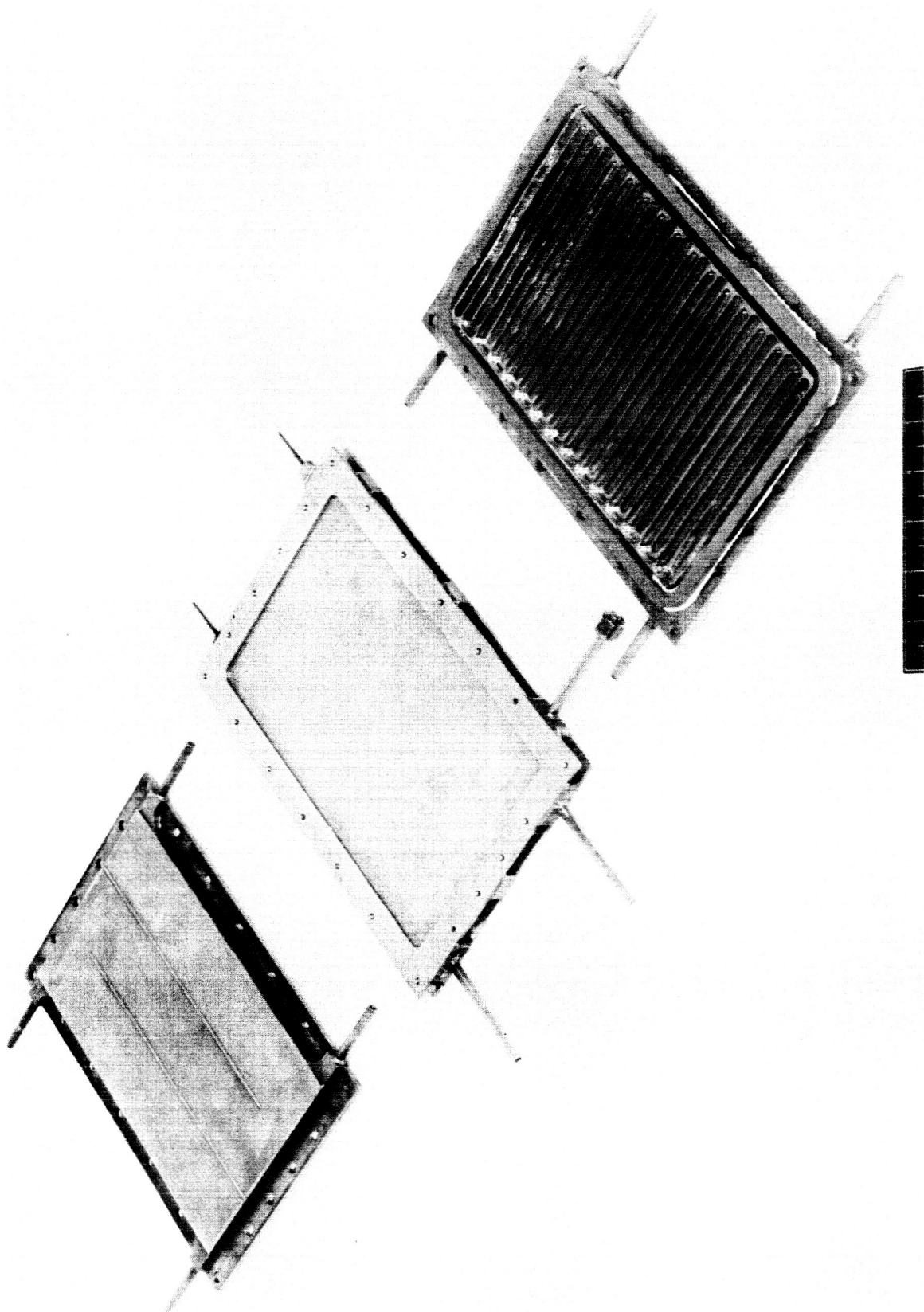


FIGURE 6. DIFFUSION STILL MODULE - DISASSEMBLED

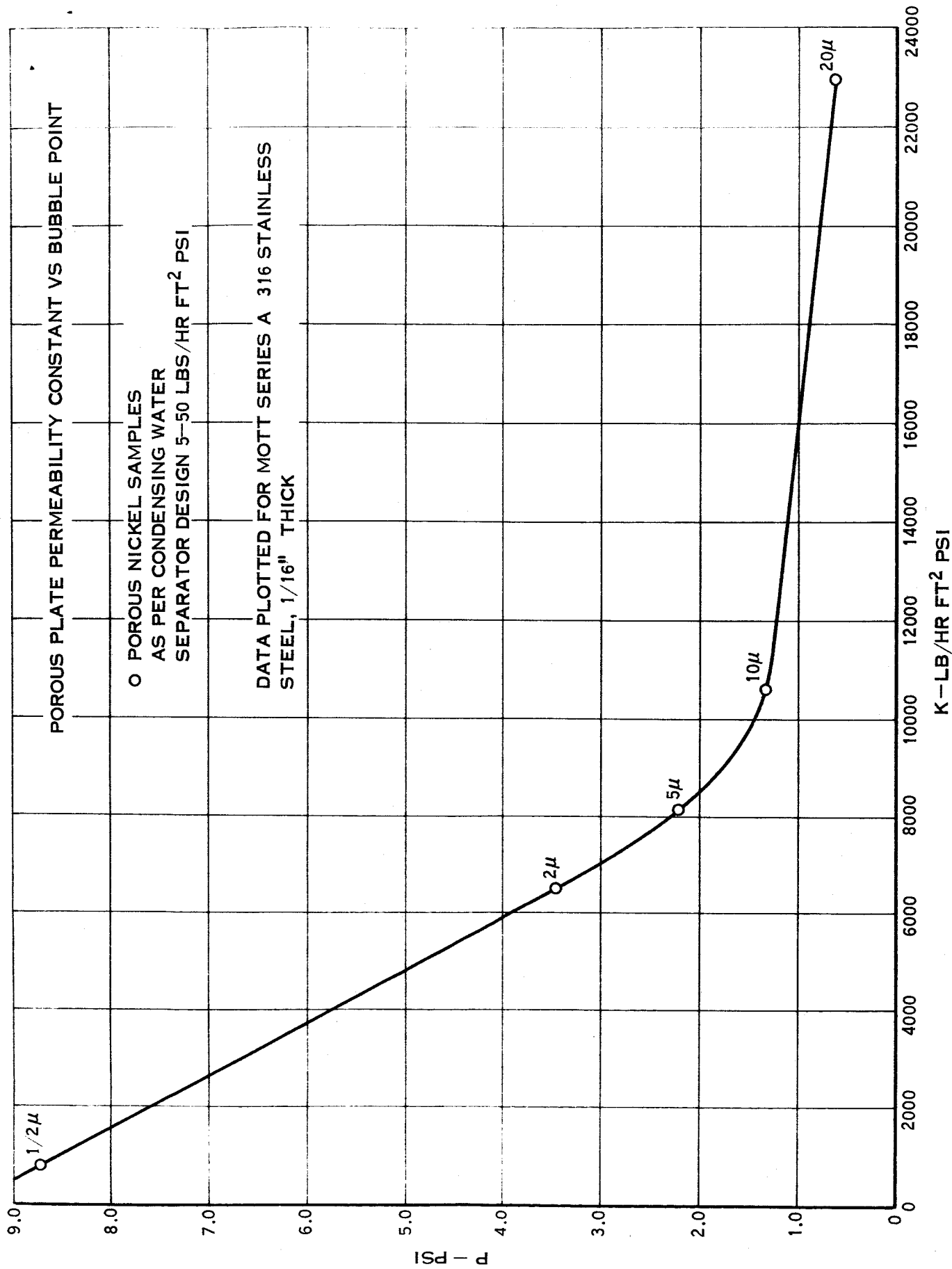


FIGURE 7. POROUS PLATE PERFORMANCE PARAMETERS

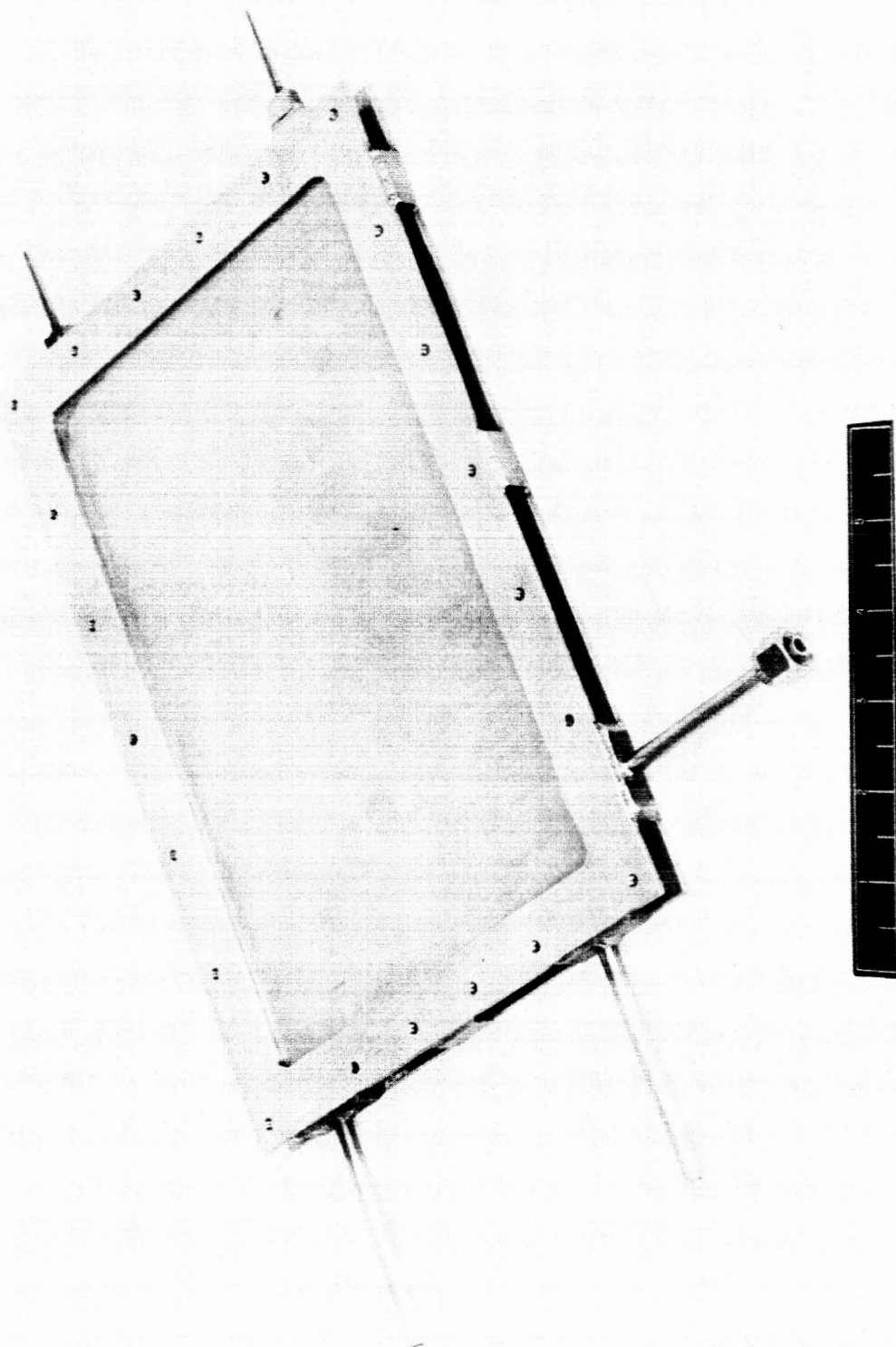


FIGURE 8. POROUS PLATE CONDENSING WATER SEPARATOR

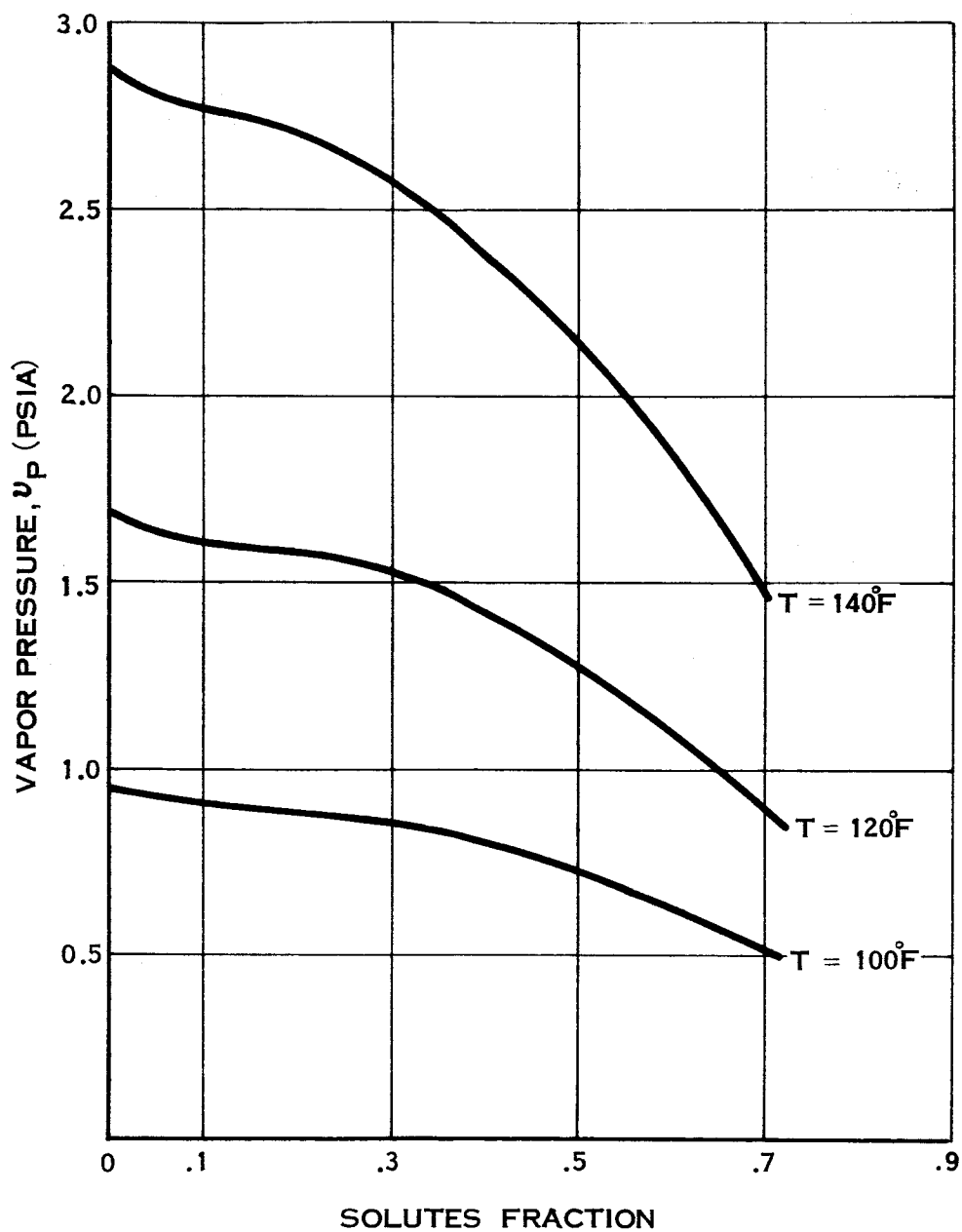


FIGURE 9. VAPOR PRESSURE OF WATER IN URINE AS A FUNCTION OF SOLIDS FRACTION AND TEMPERATURE

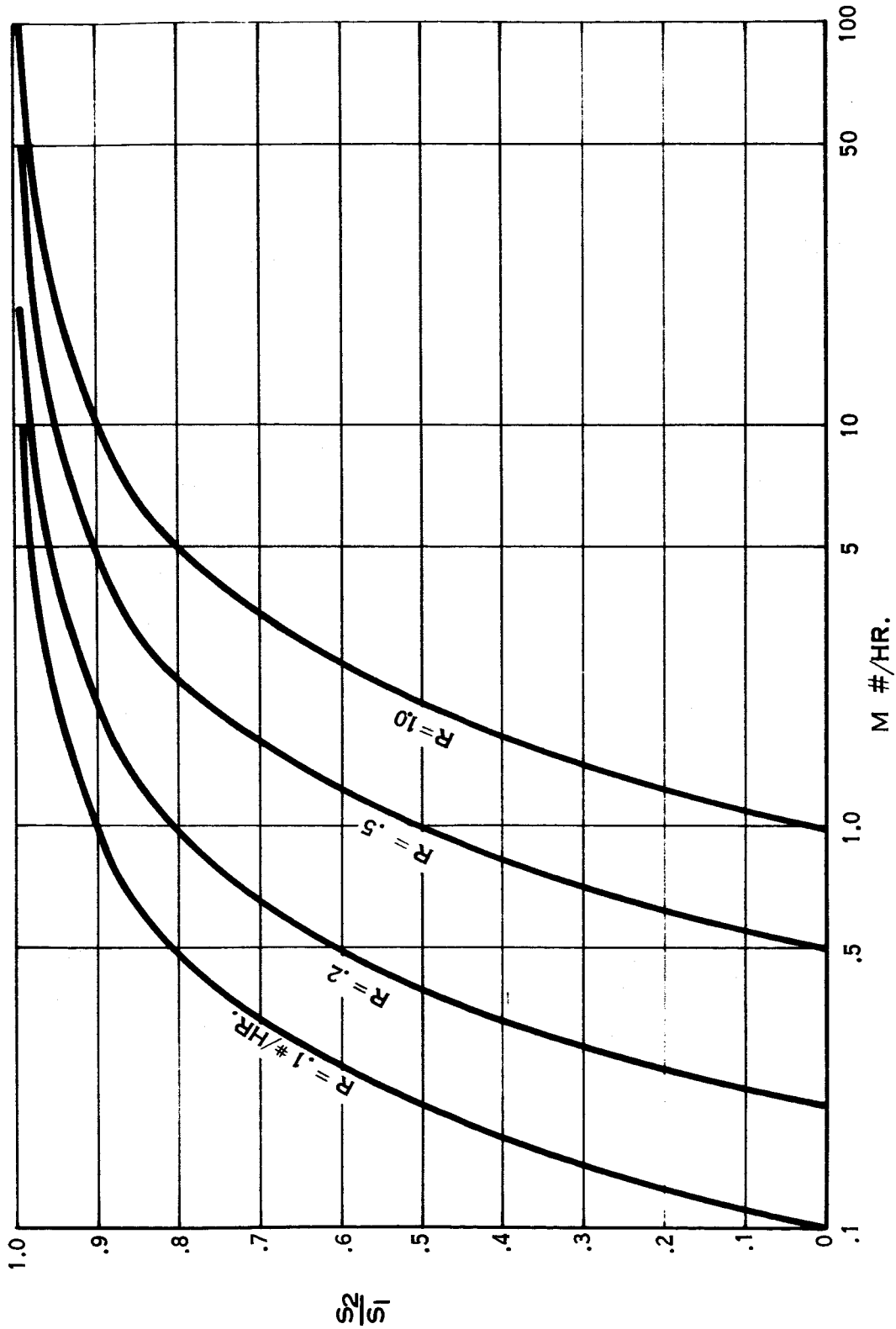


FIGURE 10. RATIO OF OUTLET TO INLET SOLIDS FRACTION AS A FUNCTION OF BRINE FLOW RATE AND WATER EXTRACTION RATE

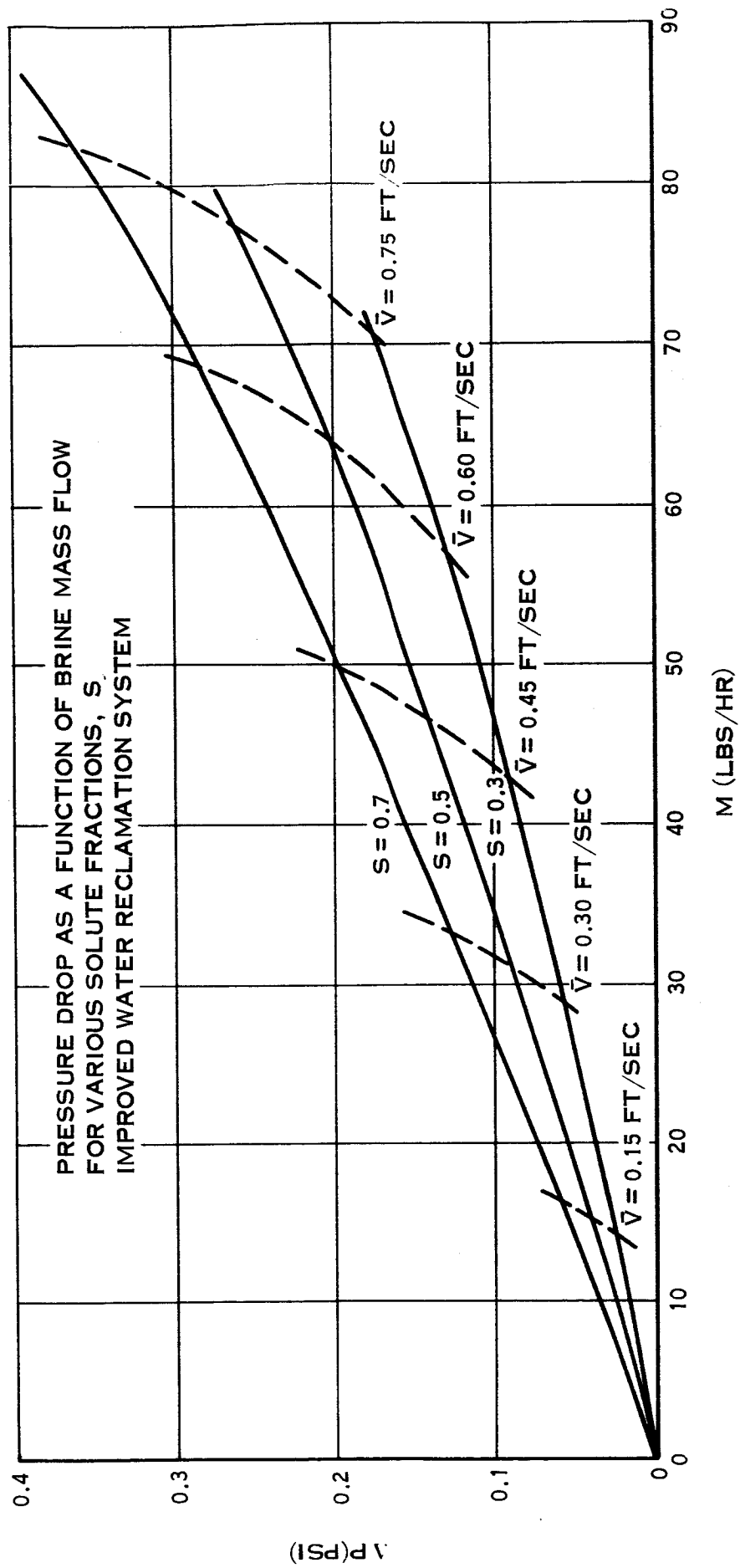


FIGURE 11. BRINE PRESSURE DROP PER HALF CELL AS A FUNCTION OF BRINE
FLOW AND SOLIDS FRACTION

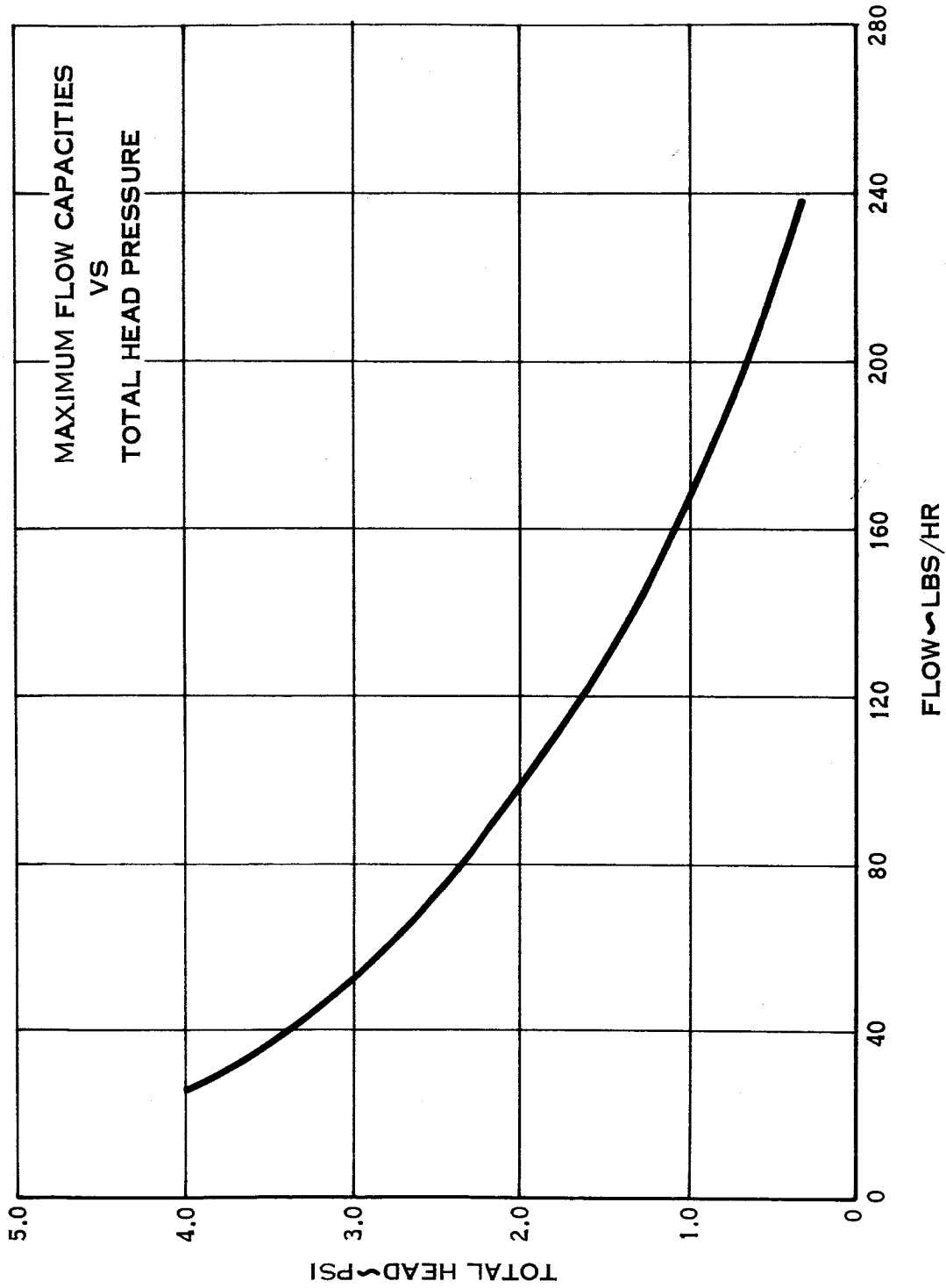


FIGURE 12. BRINE PUMP PERFORMANCE CHARACTERISTICS FOR WATER

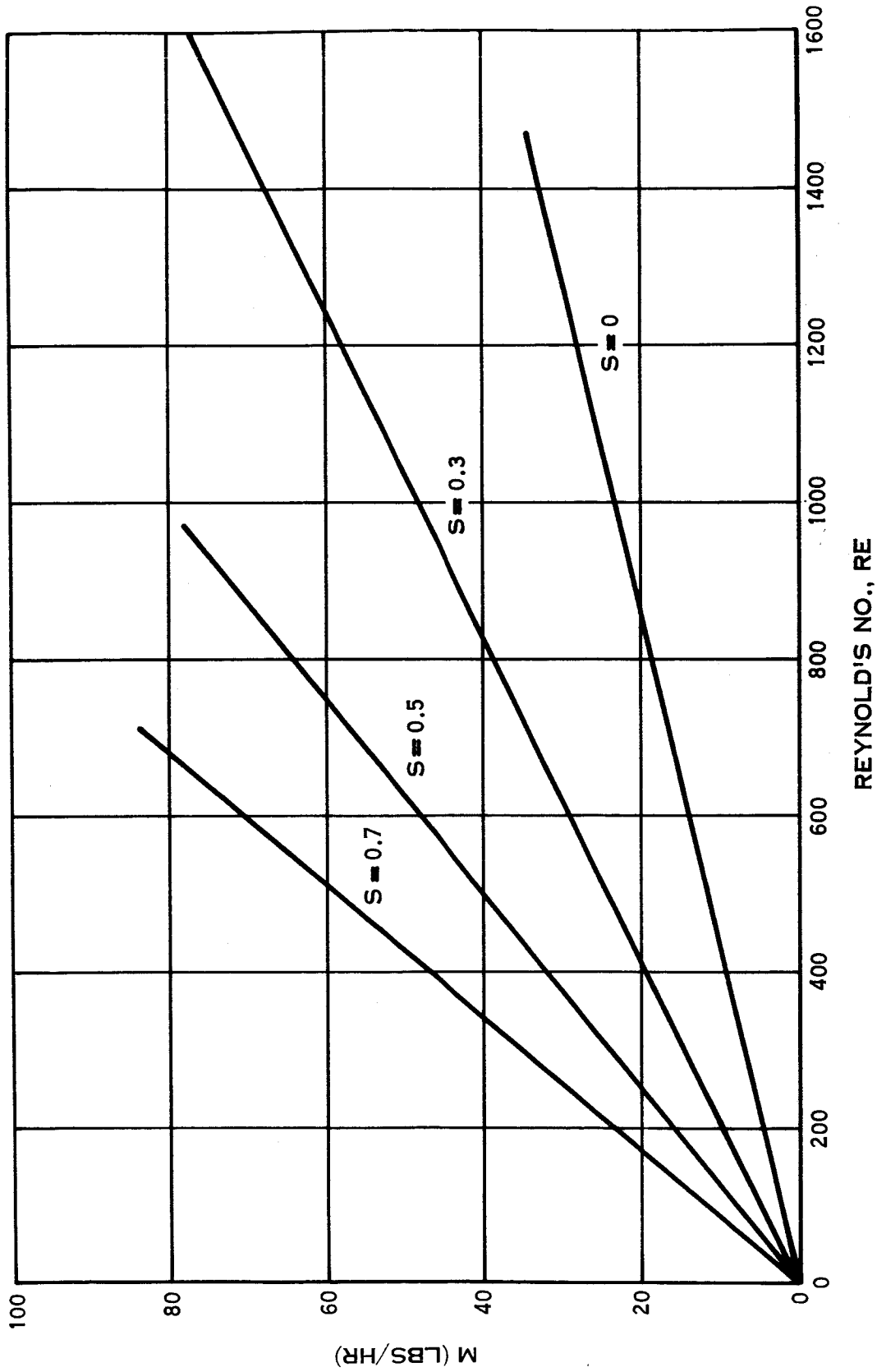


FIGURE 13. BRINE REYNOLDS NUMBER AS A FUNCTION OF BRINE FLOW AND SOLIDS FRACTION

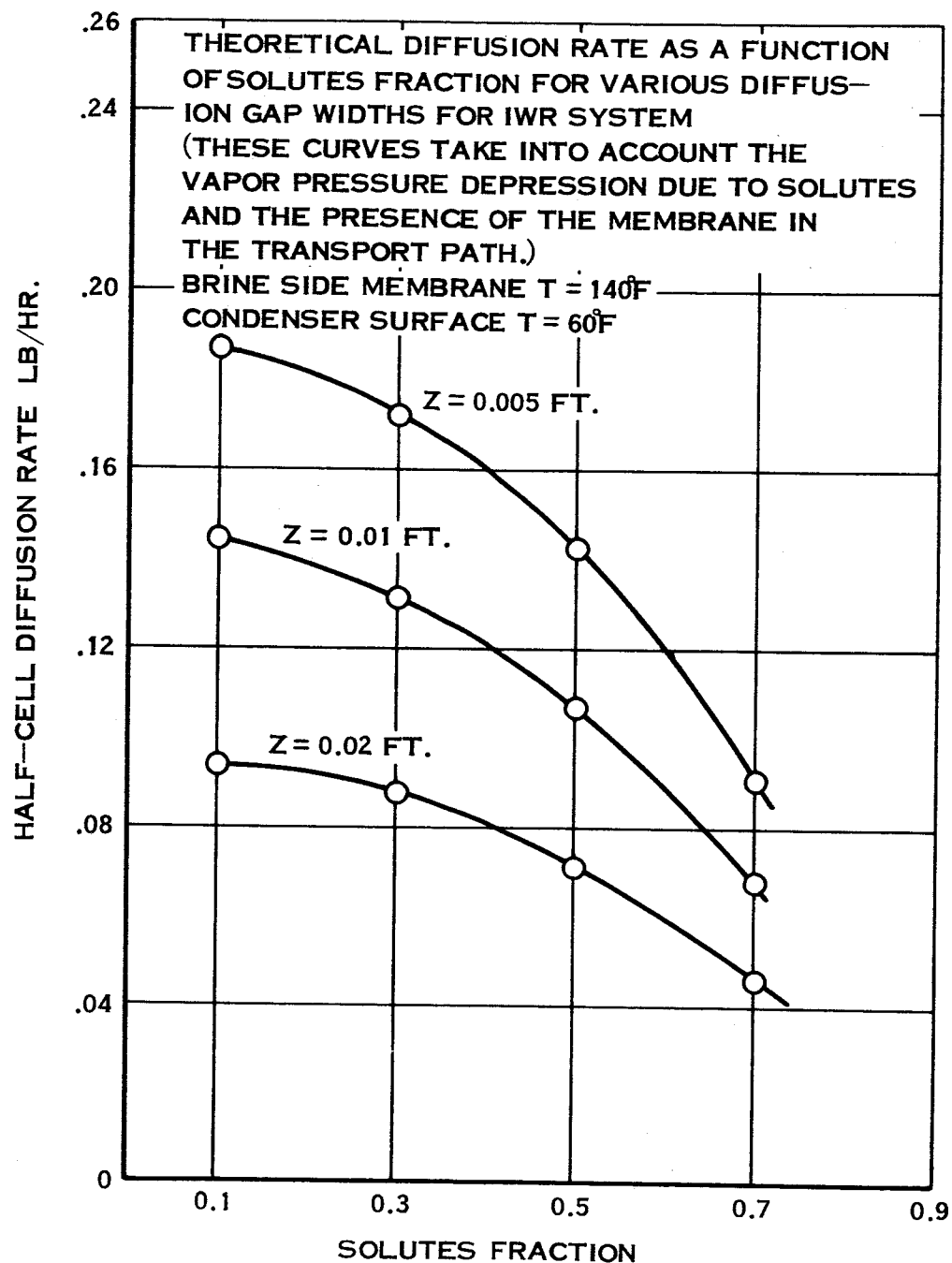


FIGURE 14. PREDICTED PROCESS RATE PER HALF CELL AS A FUNCTION OF DIFFUSION GAP WIDTH AND BRINE SOLIDS FRACTION

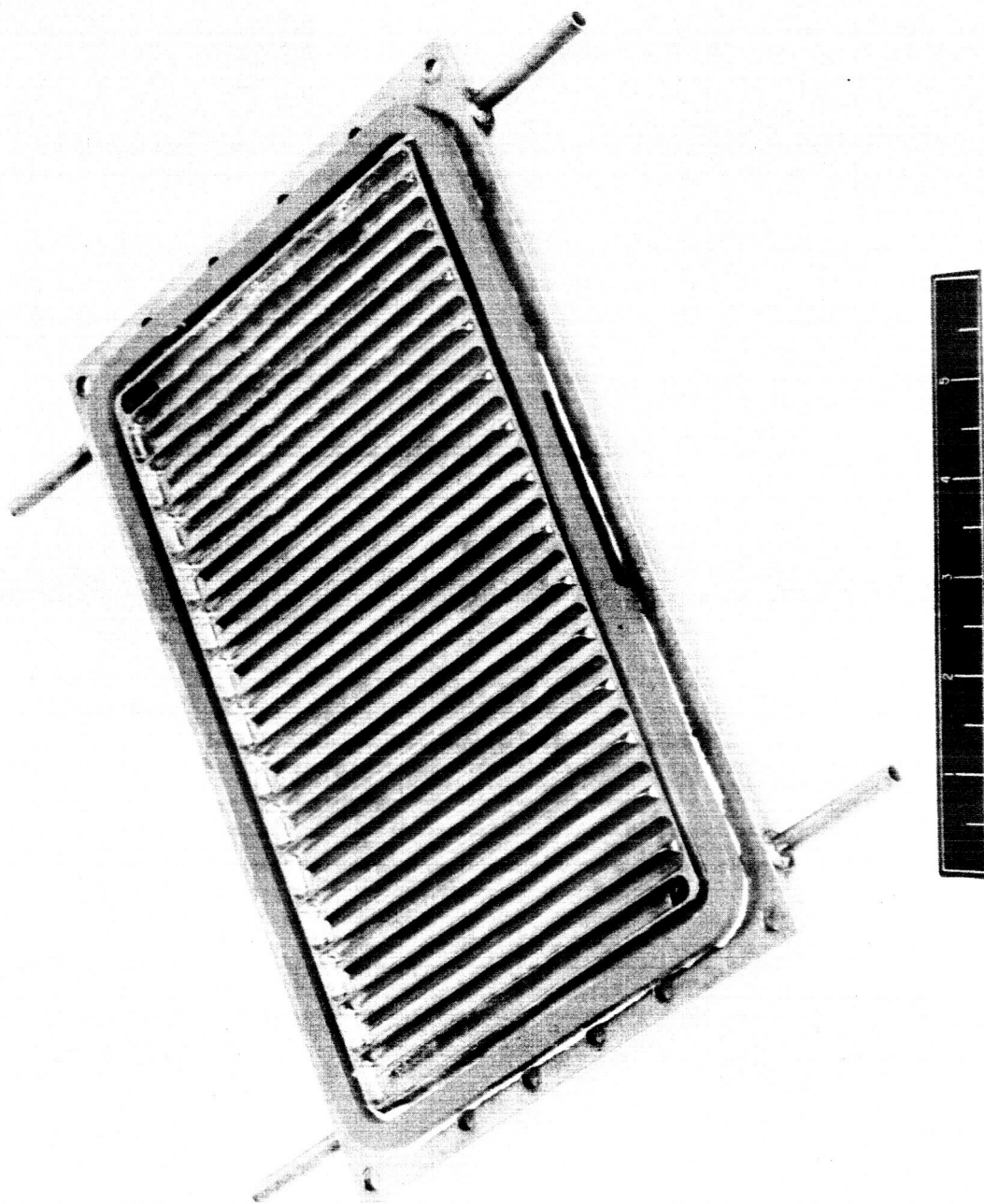
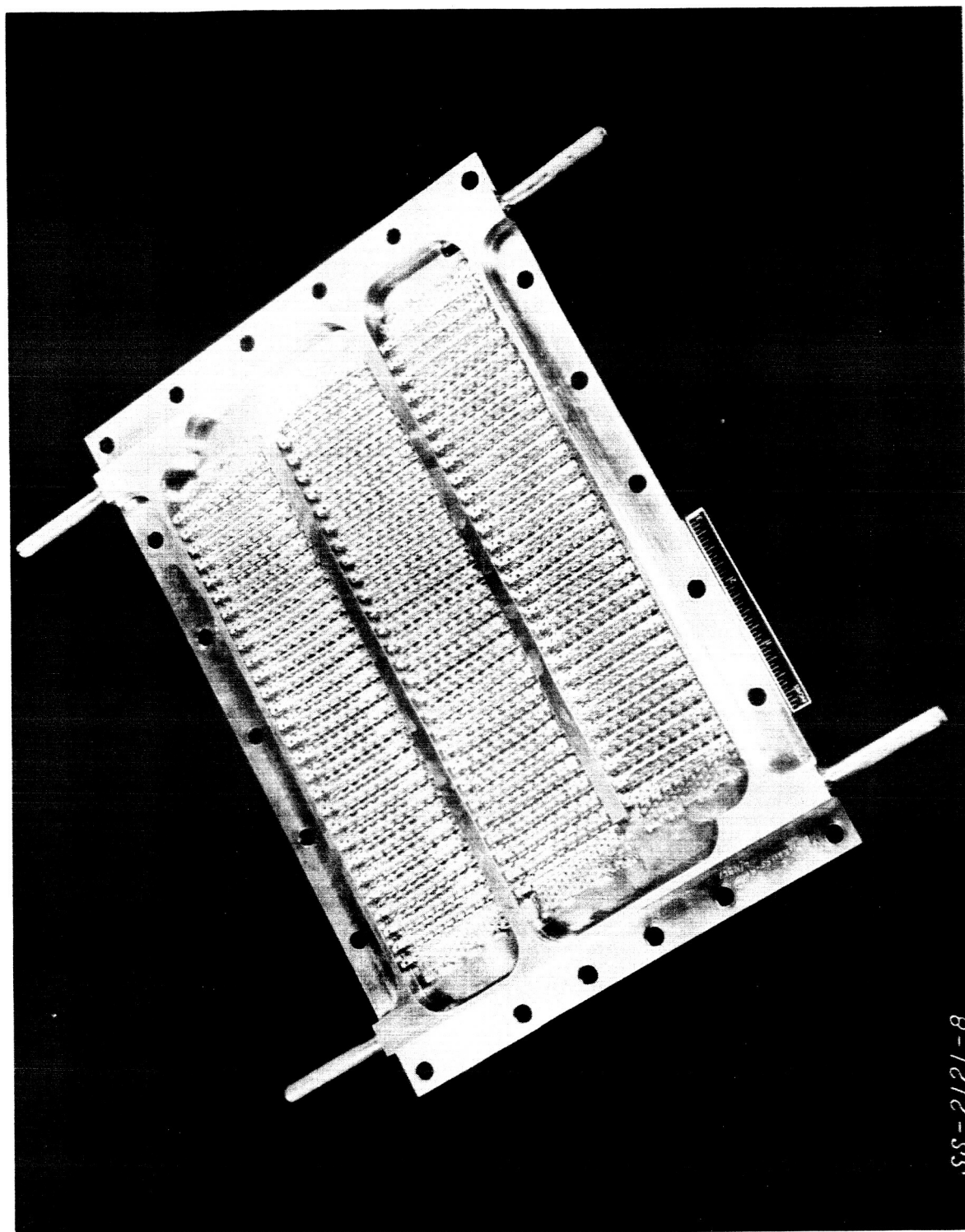


FIGURE 15. EVAPORATOR — BRINE FLOW SIDE WITH THERMAL GASKET



SS-2/21-8

FIGURE 16. EVAPORATOR — HOT FLUID SIDE WITH PERFORATED FIN
STOCK



FIGURE 17. MEMBRANE TESTING FIXTURE — DISASSEMBLED

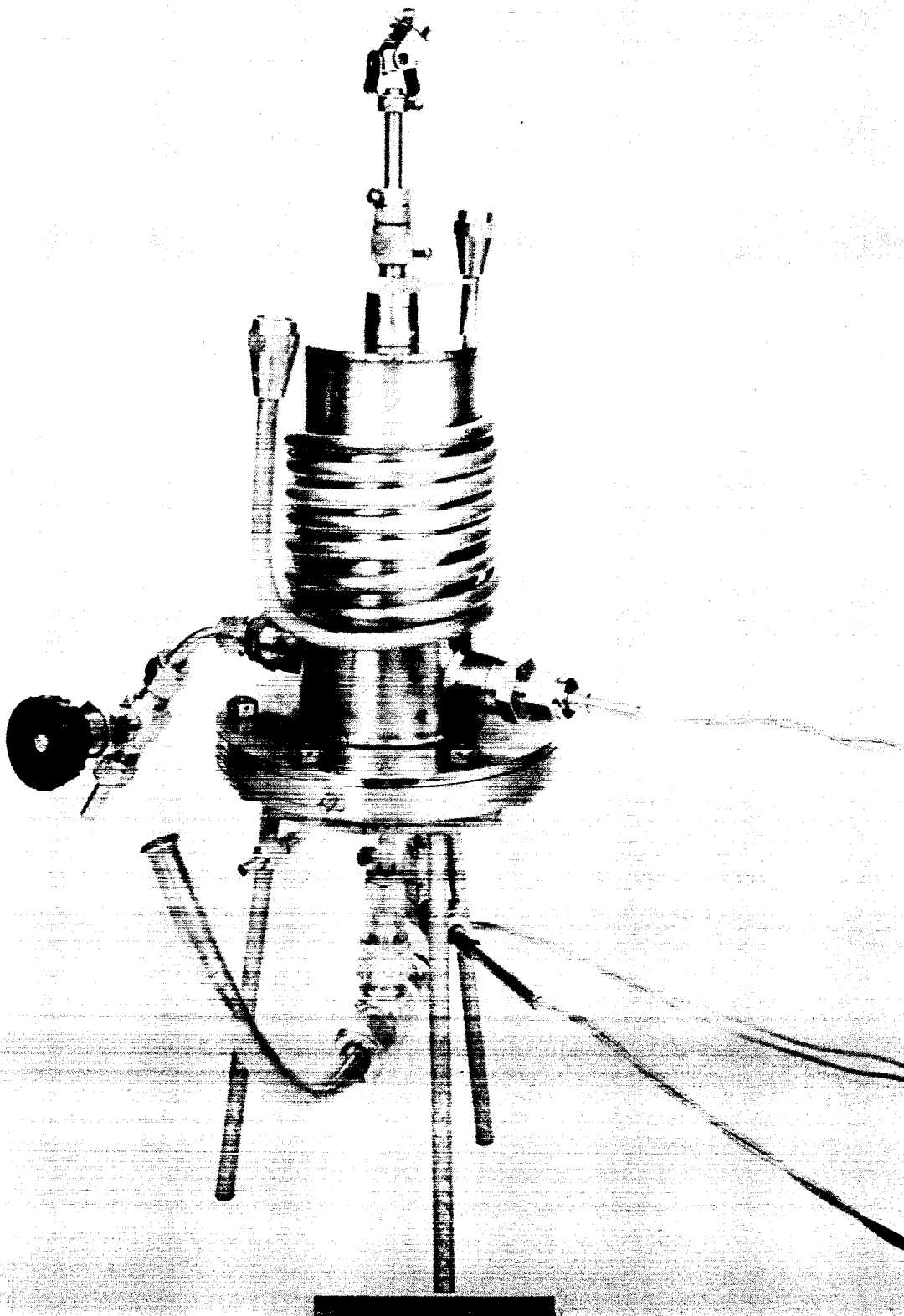


FIGURE 18. MEMBRANE TESTING FIXTURE — ASSEMBLED

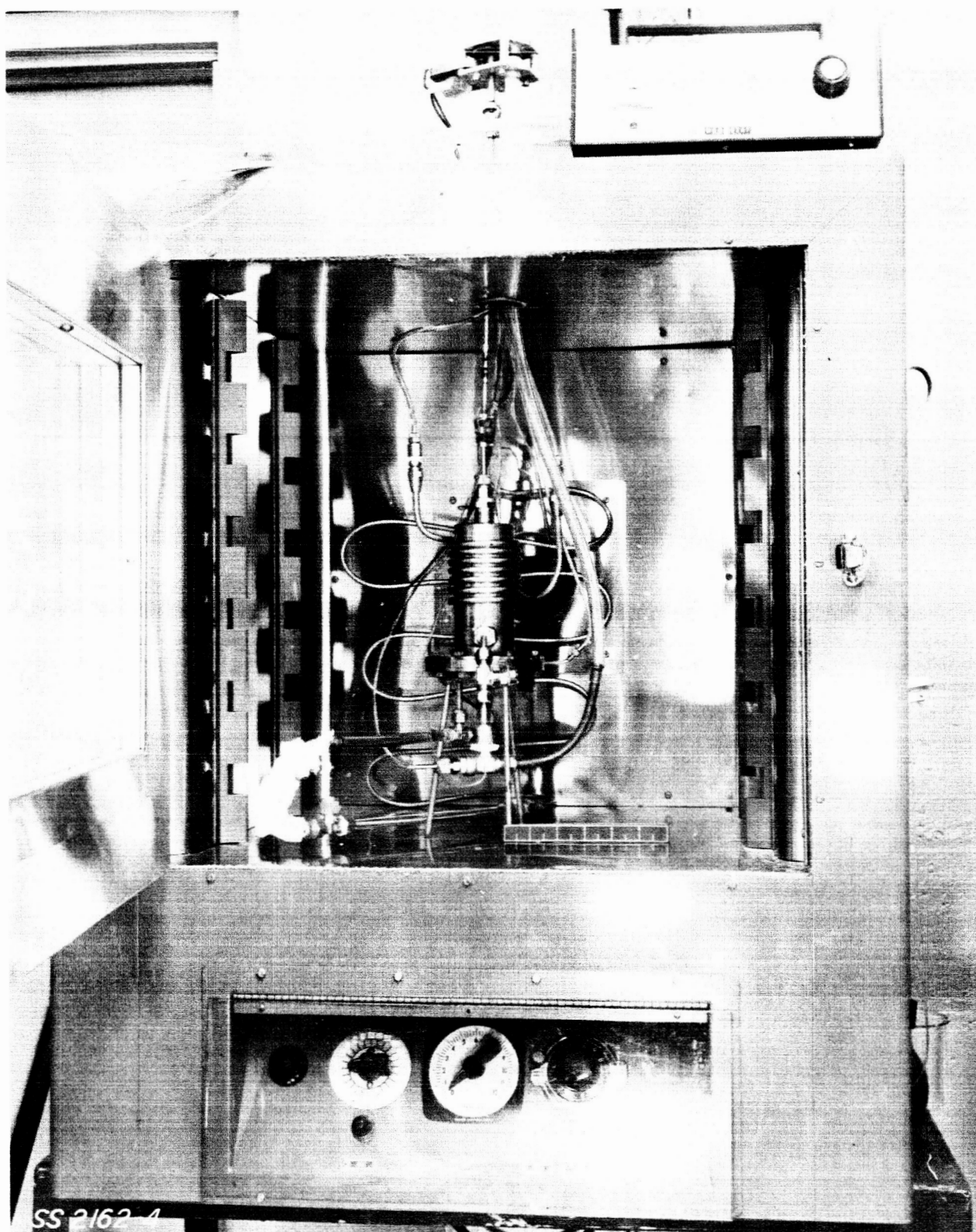


FIGURE 19. MEMBRANE TESTING FIXTURE — TEMPERATURE
CONTROLLED FACILITY



FIGURE 20. MEMBRANE TEST FACILITY

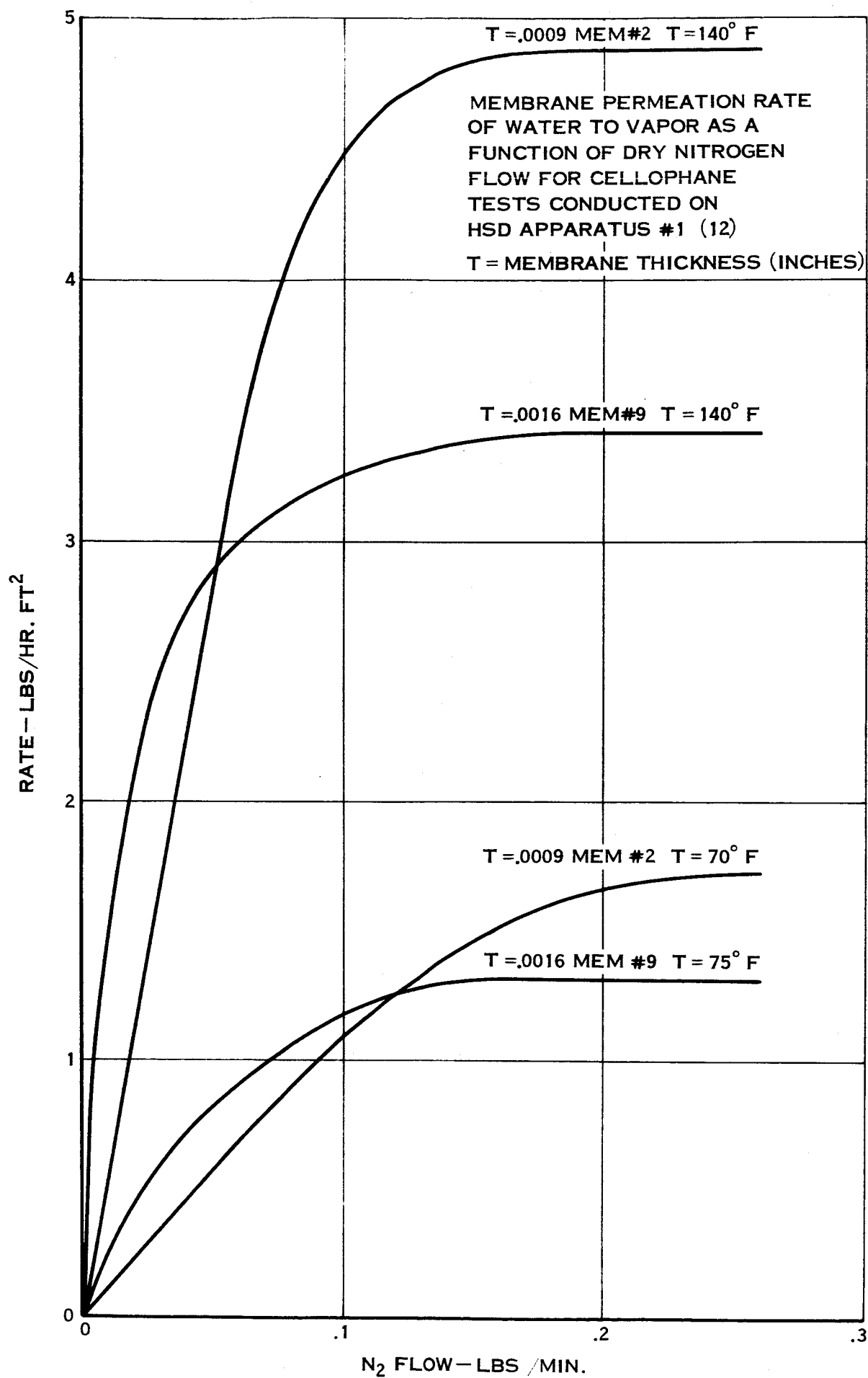


FIGURE 21. PERMEATION RATE OF WATER FOR CELLOPHANE AS A FUNCTION OF THICKNESS

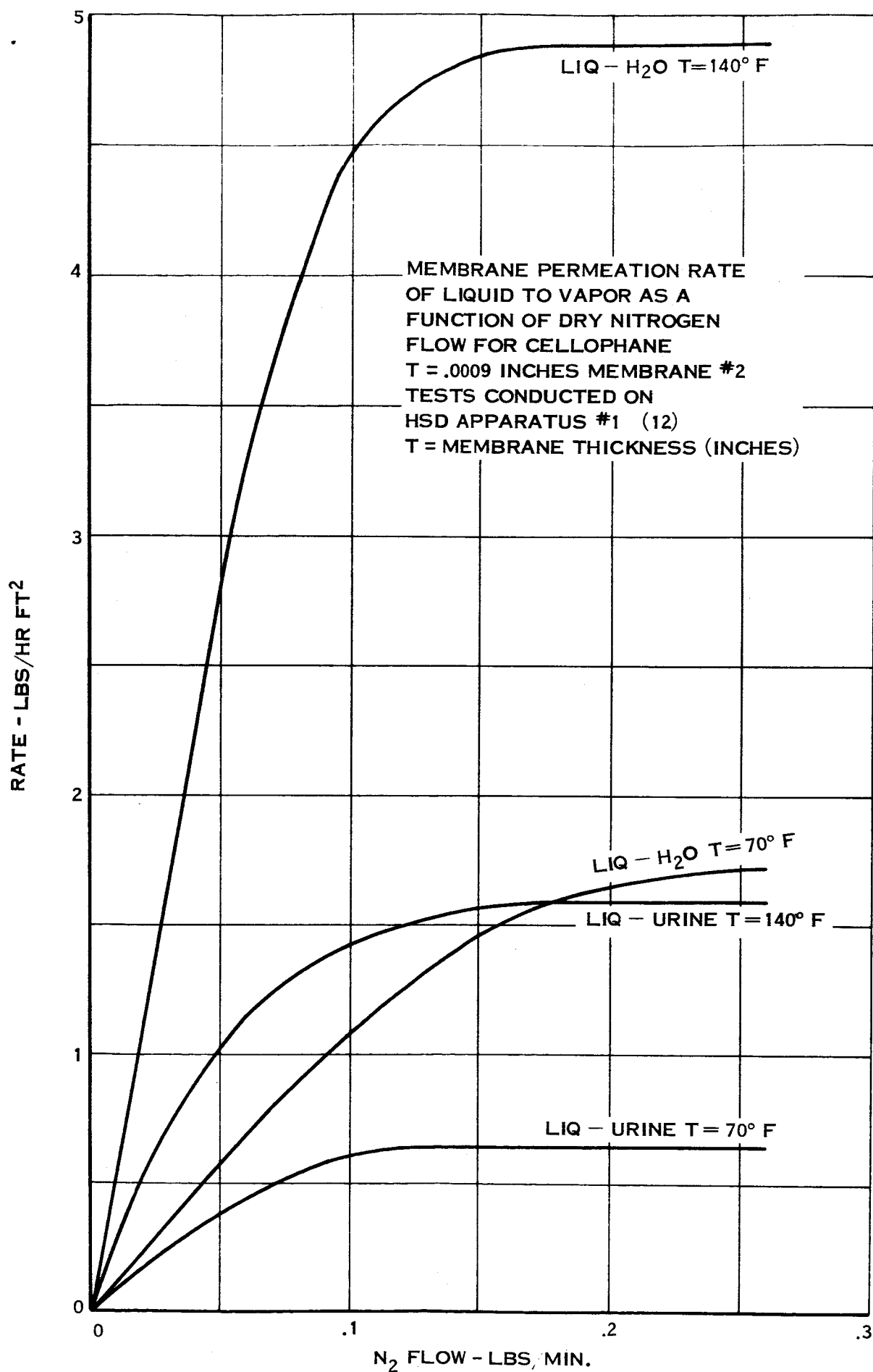


FIGURE 22. PERMEATION RATE OF LIQUID FOR CELLOPHANE WITH URINE AND WATER

TEST LIQUID - WATER

- ① T = .0009 MEM #4 HSD APPARATUS #1 (12)
- ② T = .0009 MEM #2 HSD APPARATUS #1 (12)
- ③ T = .0016 MEM #9 HSD APPARATUS #1 (12)
- ④ T = .0013 HSD APPARATUS #2 (13)

TEST LIQUID - URINE

- ⑤ T = .0009 MEM #2 HSD APPARATUS #1 (12)
- ⑥ T = .0009 MEM #4 HSD APPARATUS #1 (12)
- ⑦ T = .0013 WARNER, BROWN, GLASS (7)

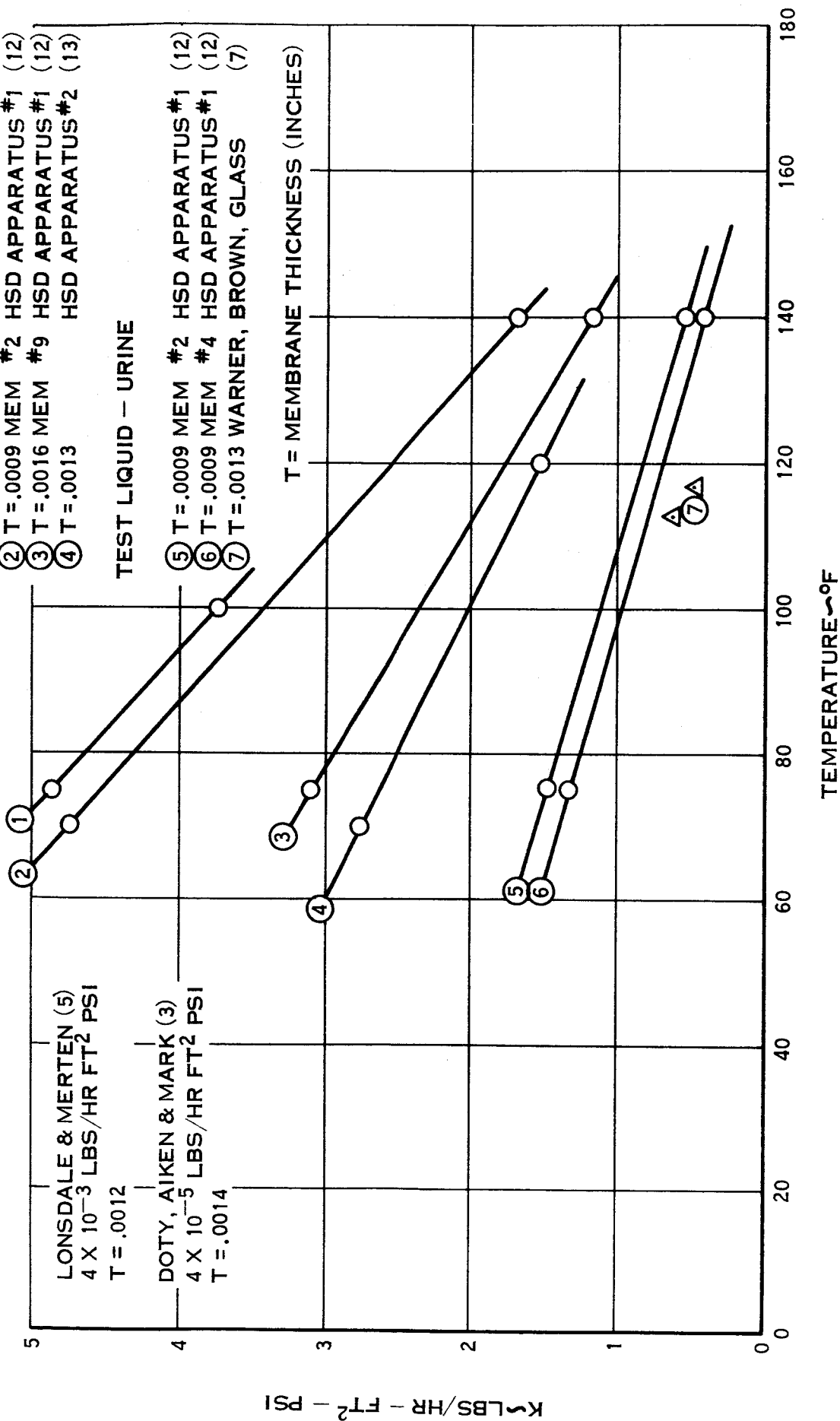


FIGURE 23. PERMEABILITY CONSTANT FOR CELLOPHANE AS A FUNCTION OF TEMPERATURE

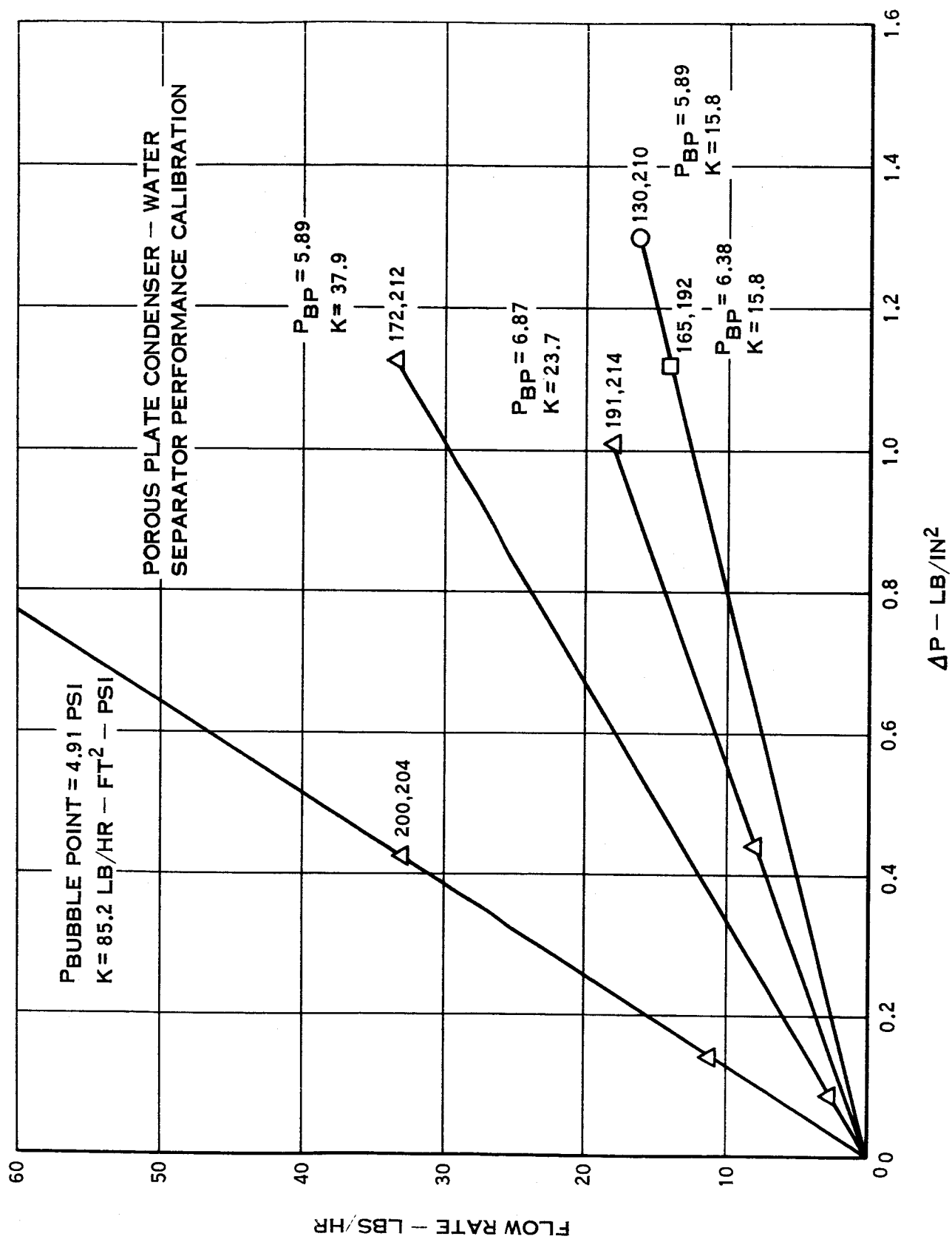


FIGURE 24. POROUS PLATE CONDENSER-WATER SEPARATOR PERFORMANCE CALIBRATION

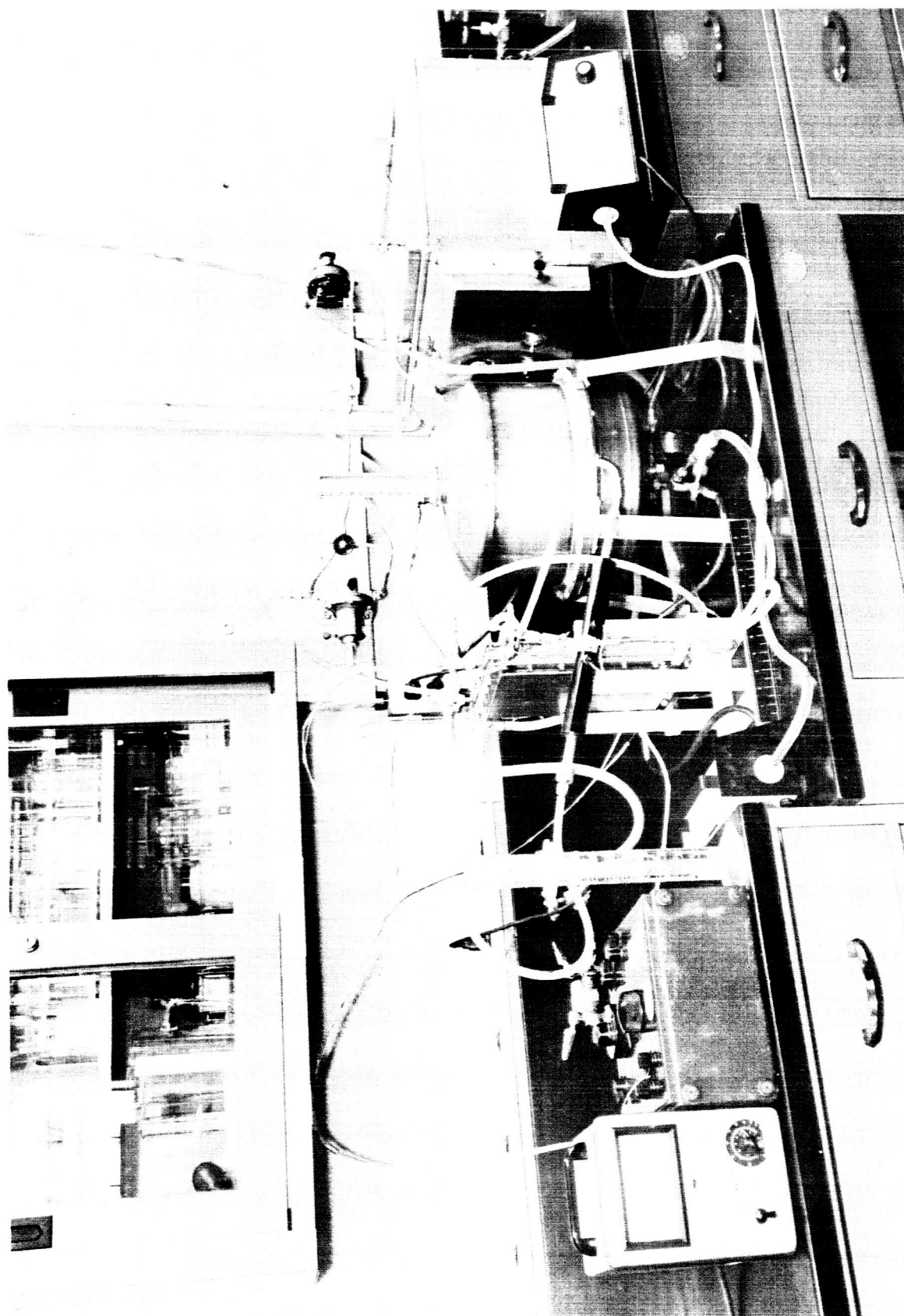
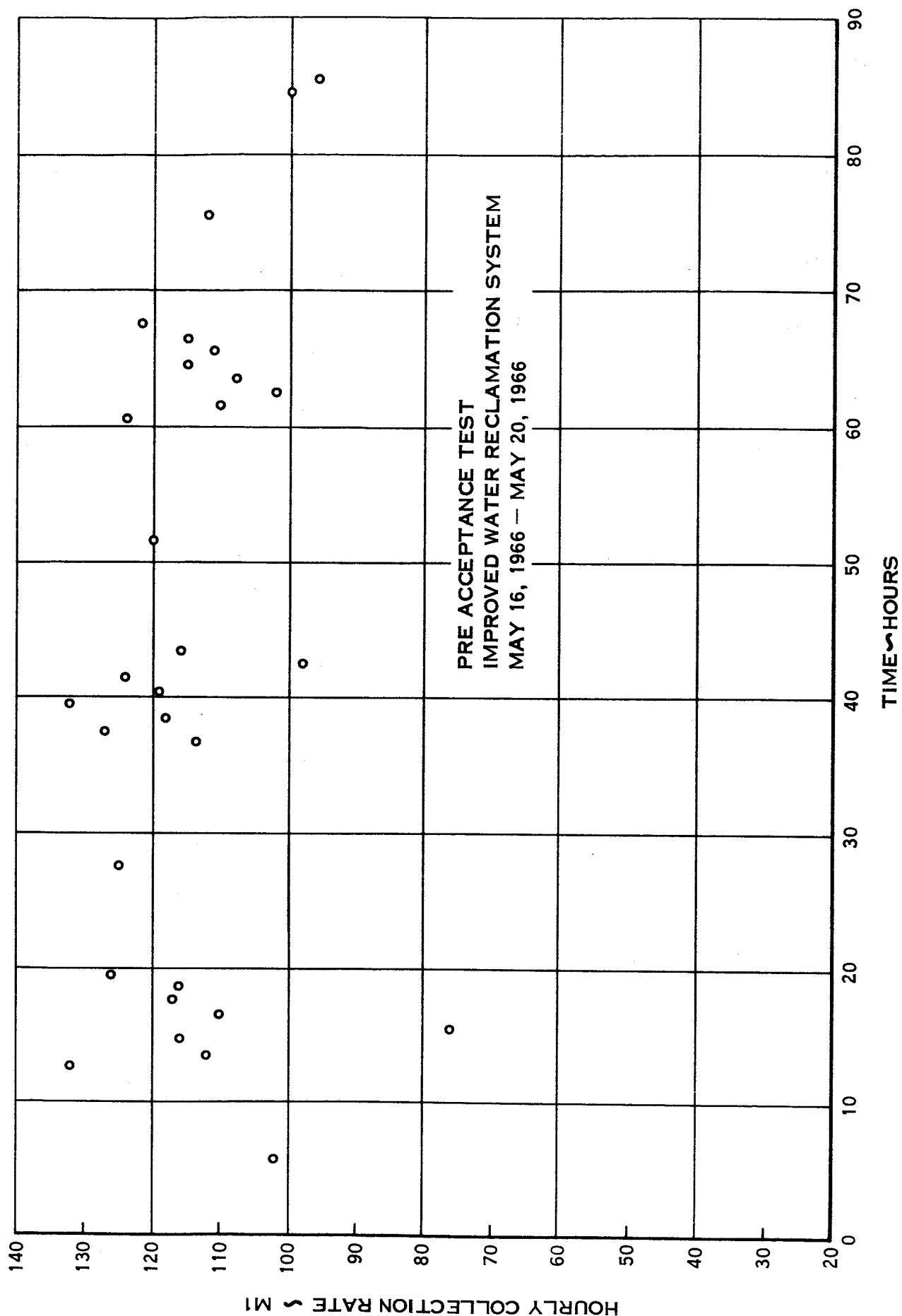


FIGURE 25. PRE-ACCEPTANCE TEST SET-UP

FIGURE 26. PRE-ACCEPTANCE TEST PRODUCT WATER COLLECTION RATE AS A FUNCTION OF TEST DURATION



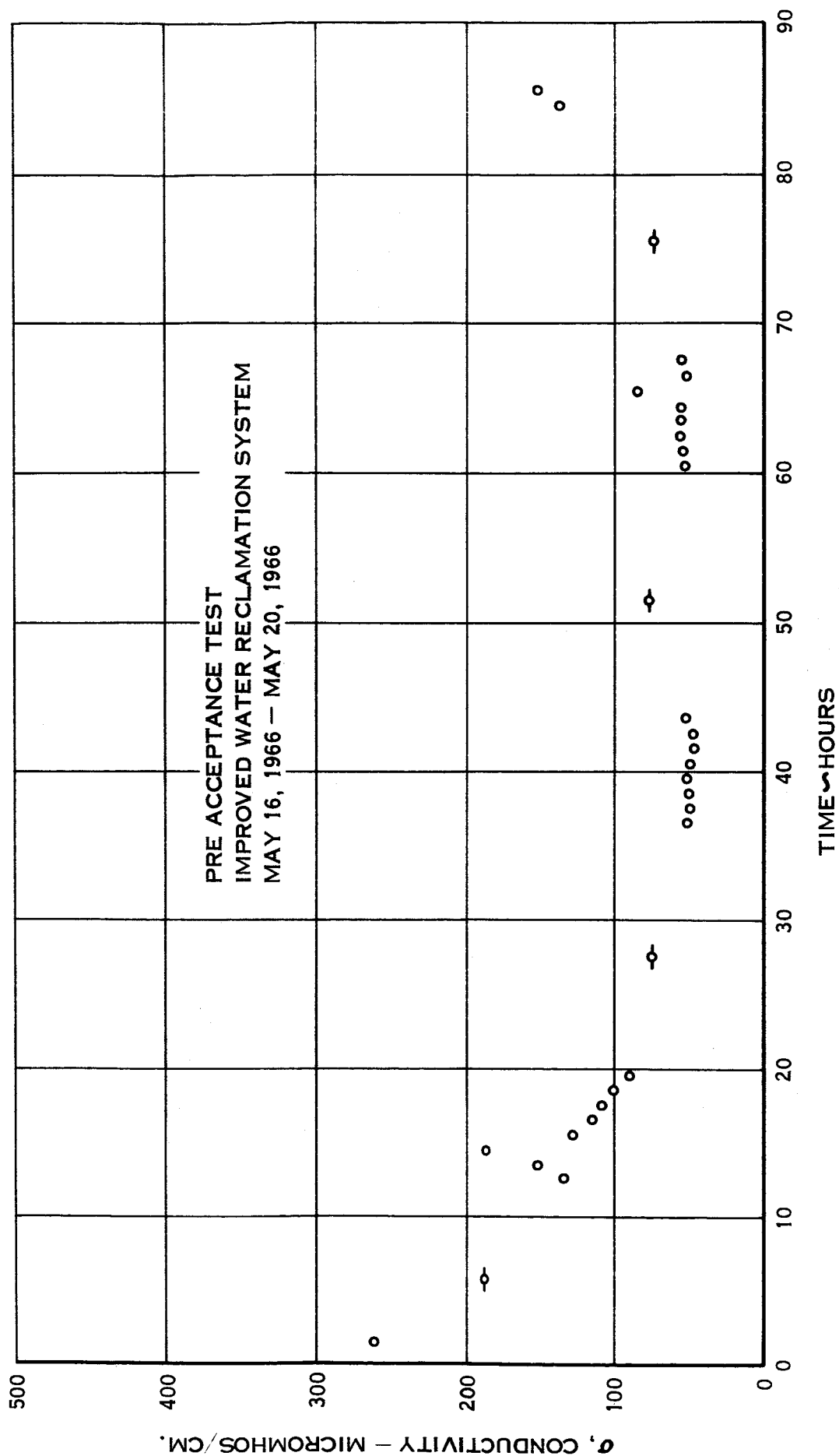


FIGURE 27. PRE-ACCEPTANCE TEST PRODUCT WATER CONDUCTIVITY AS A FUNCTION OF TEST DURATION

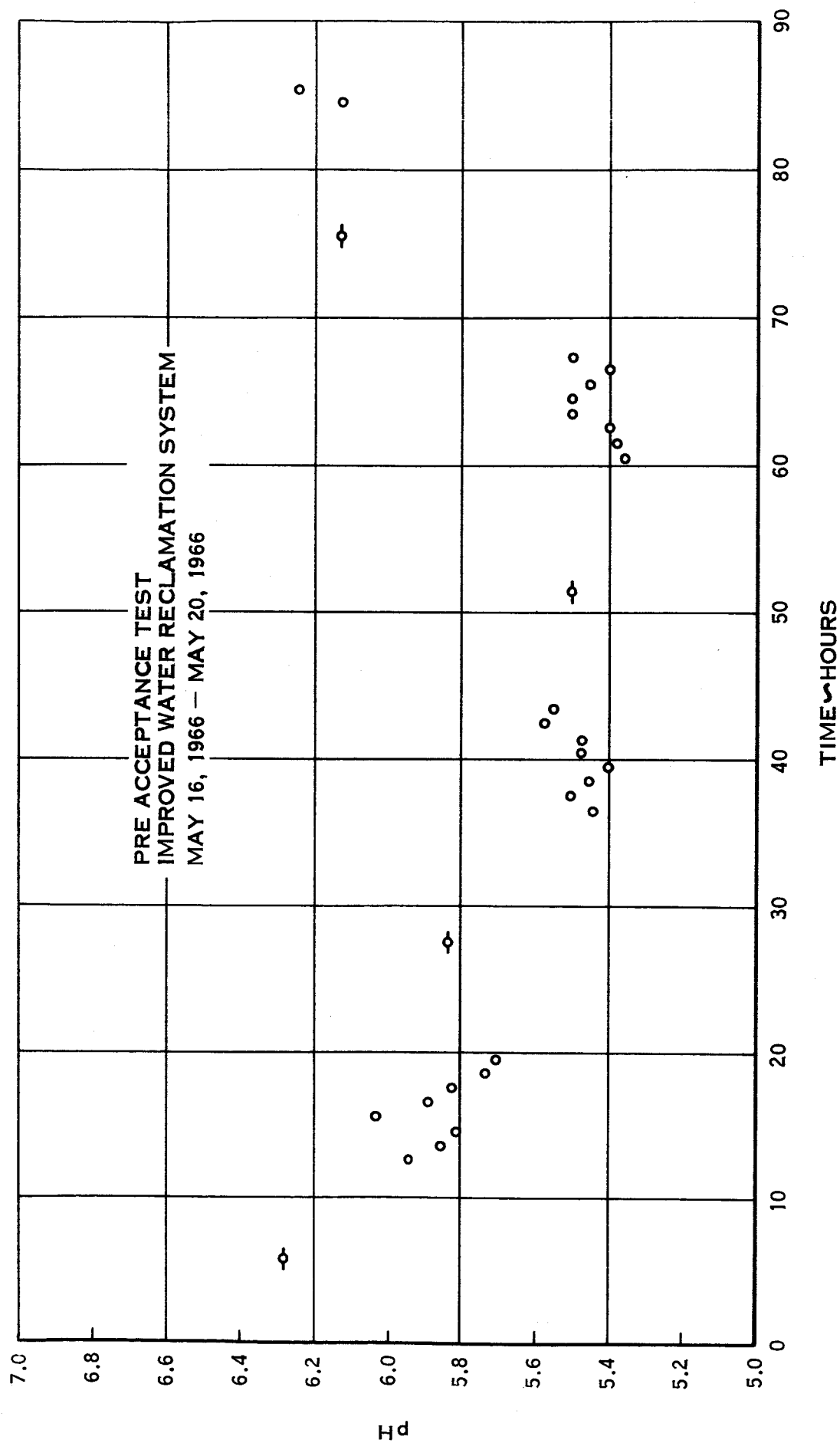


FIGURE 28. PRE-ACCEPTANCE TEST PRODUCT WATER pH AS A FUNCTION OF TEST DURATION

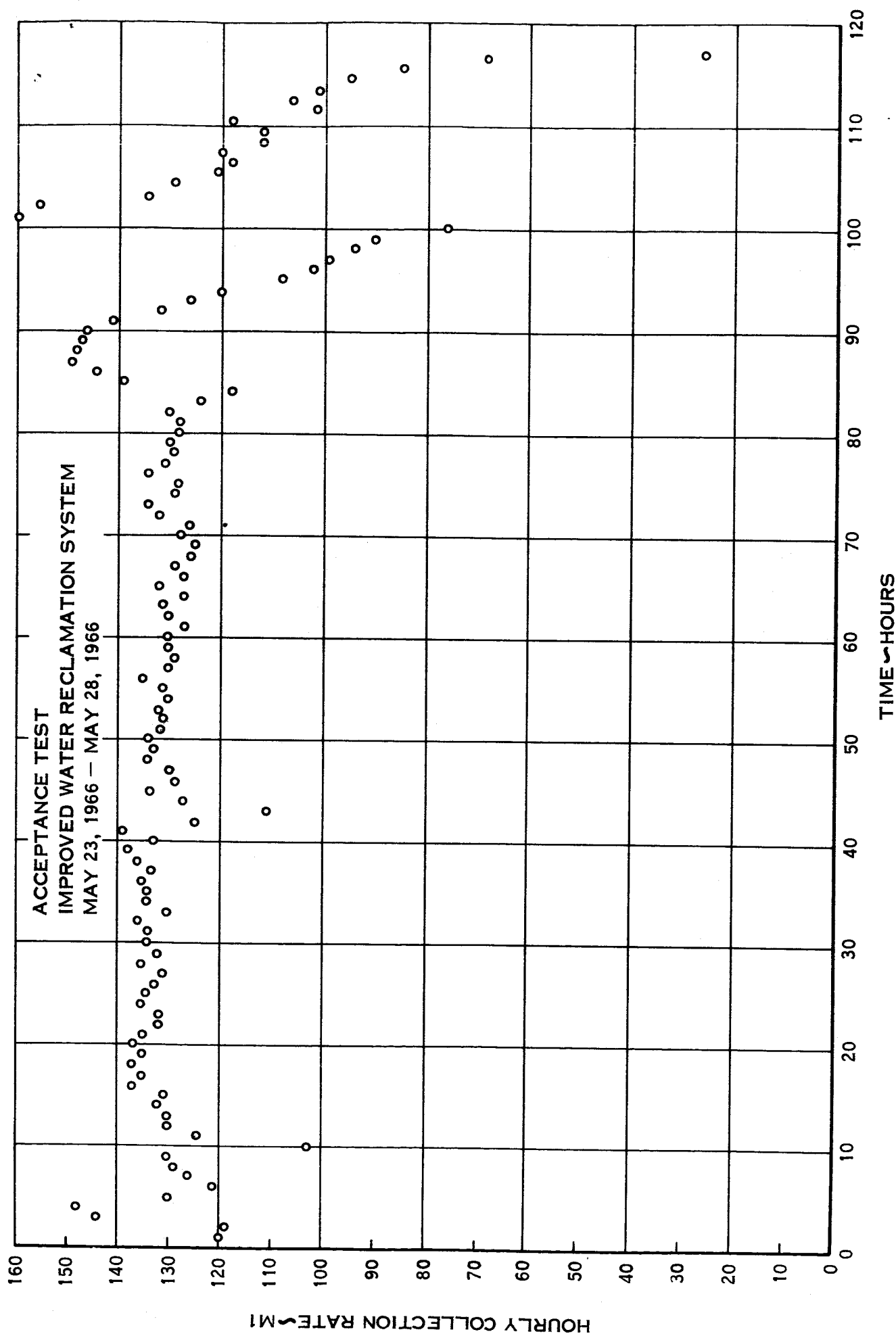


FIGURE 29. ACCEPTANCE TEST PRODUCT WATER COLLECTION RATE AS A FUNCTION OF TEST DURATION

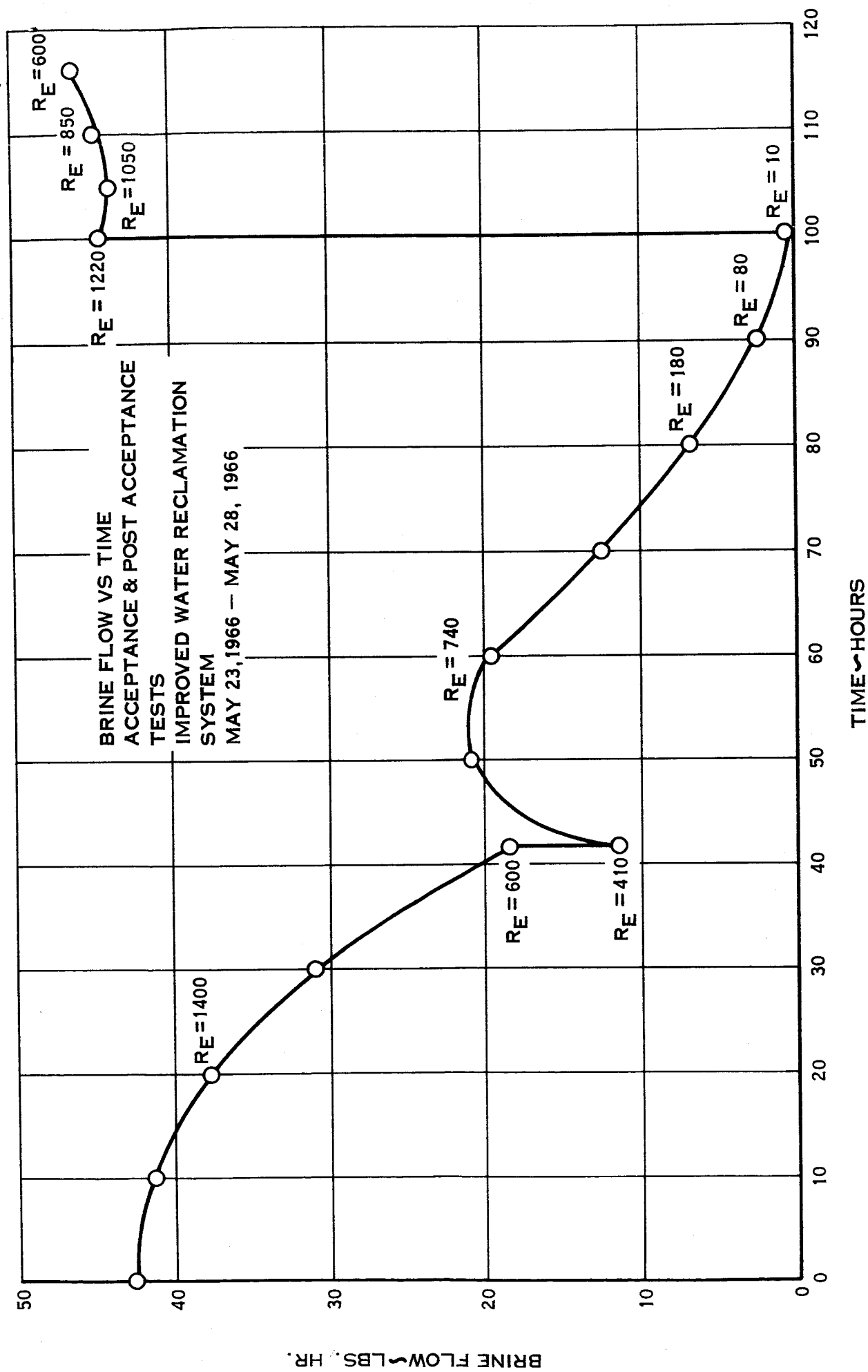


FIGURE 30. ACCEPTANCE TEST BRINE FLOW RATE AS A FUNCTION OF TEST DURATION

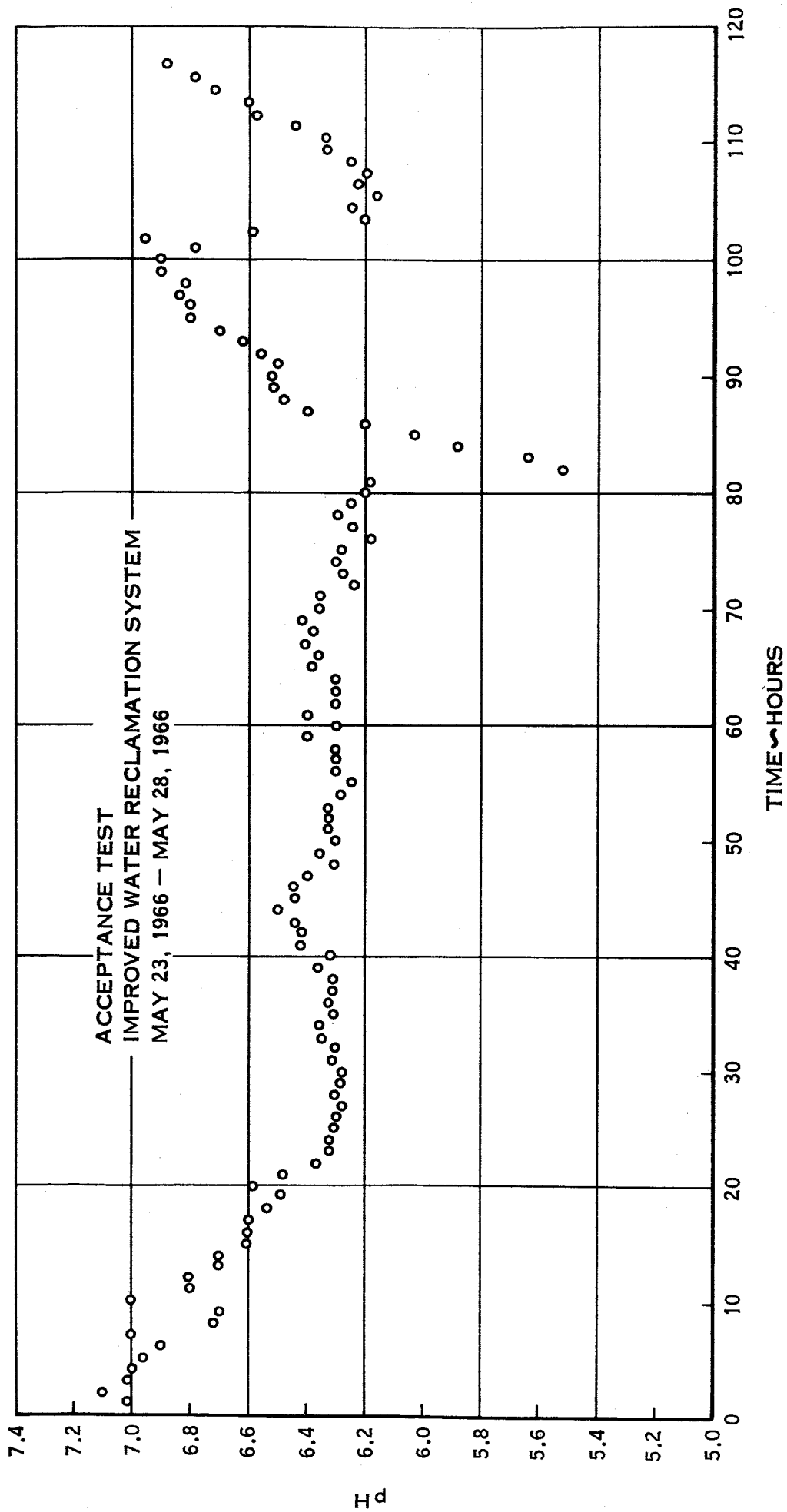


FIGURE 31. ACCEPTANCE TEST PRODUCT WATER pH AS A FUNCTION OF TEST DURATION

ACCEPTANCE TEST
IMPROVED WATER RECLAMATION SYSTEM
MAY 23, 1966 - MAY 28, 1966

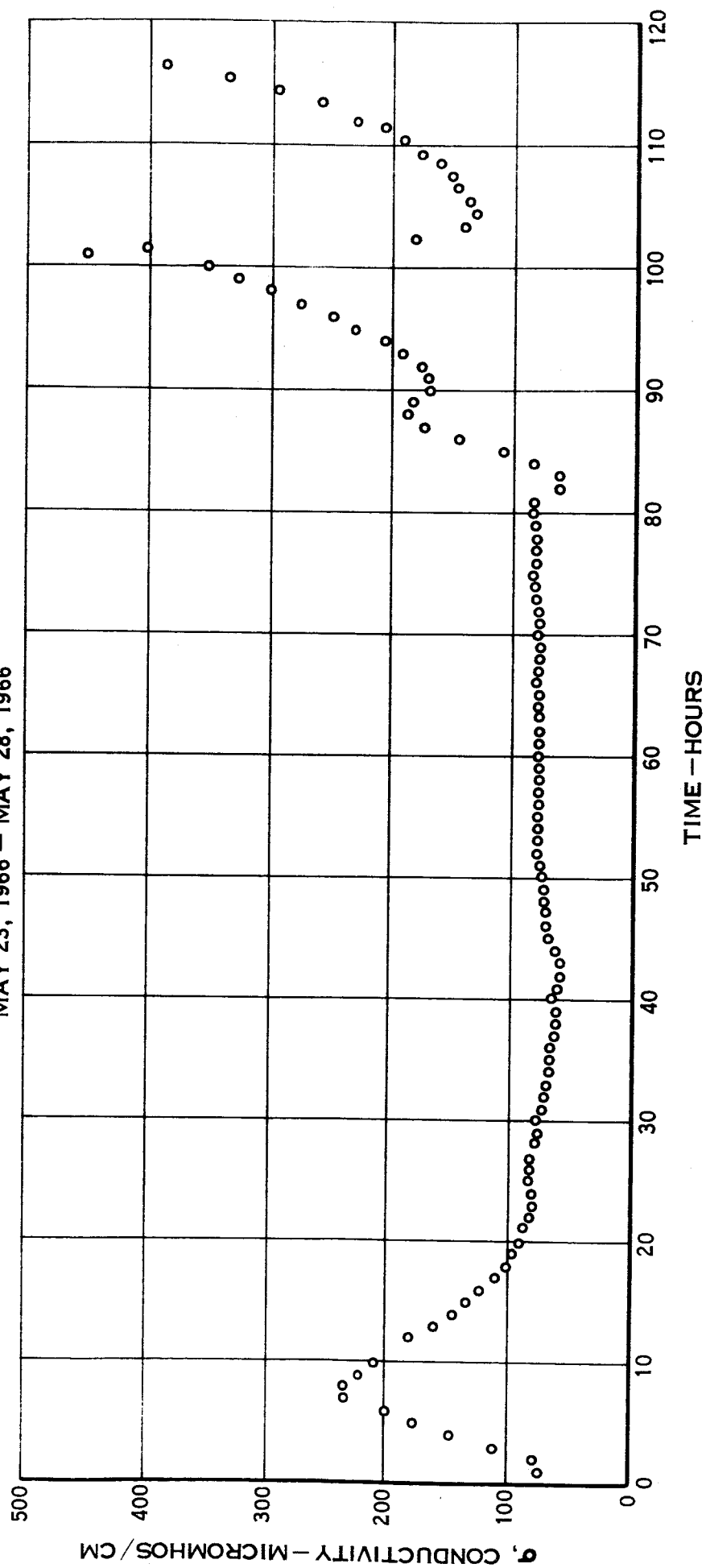


FIGURE 32. ACCEPTANCE TEST PRODUCT WATER CONDUCTIVITY AS A FUNCTION OF TEST DURATION



FIGURE 33. BATCH TANK BRINE OUTLET FITTING PLUGGED
BY GALVANIC CORROSION

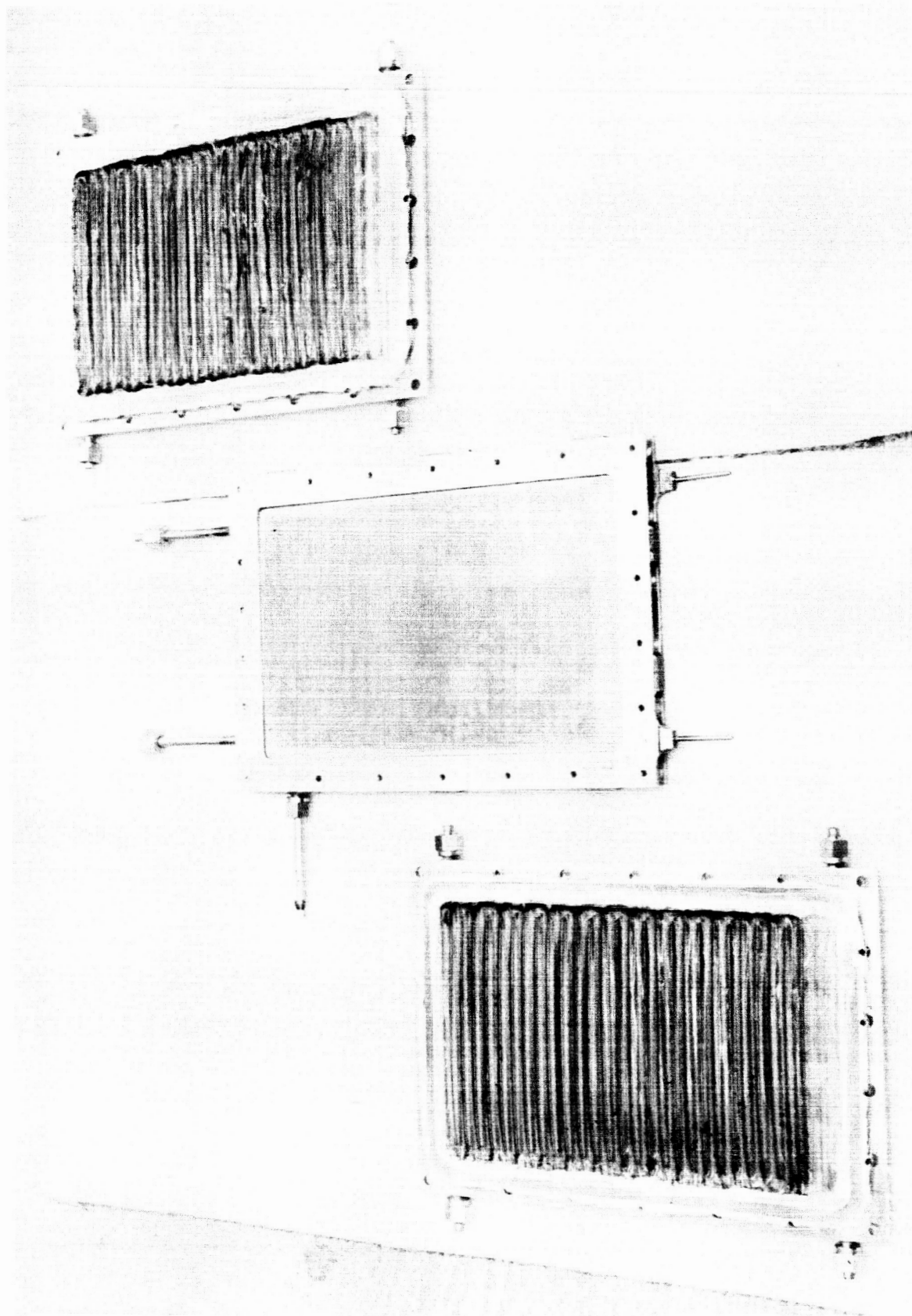


FIGURE 34. ACCEPTANCE TEST MODULE DISASSEMBLED
AT TEST CONCLUSION

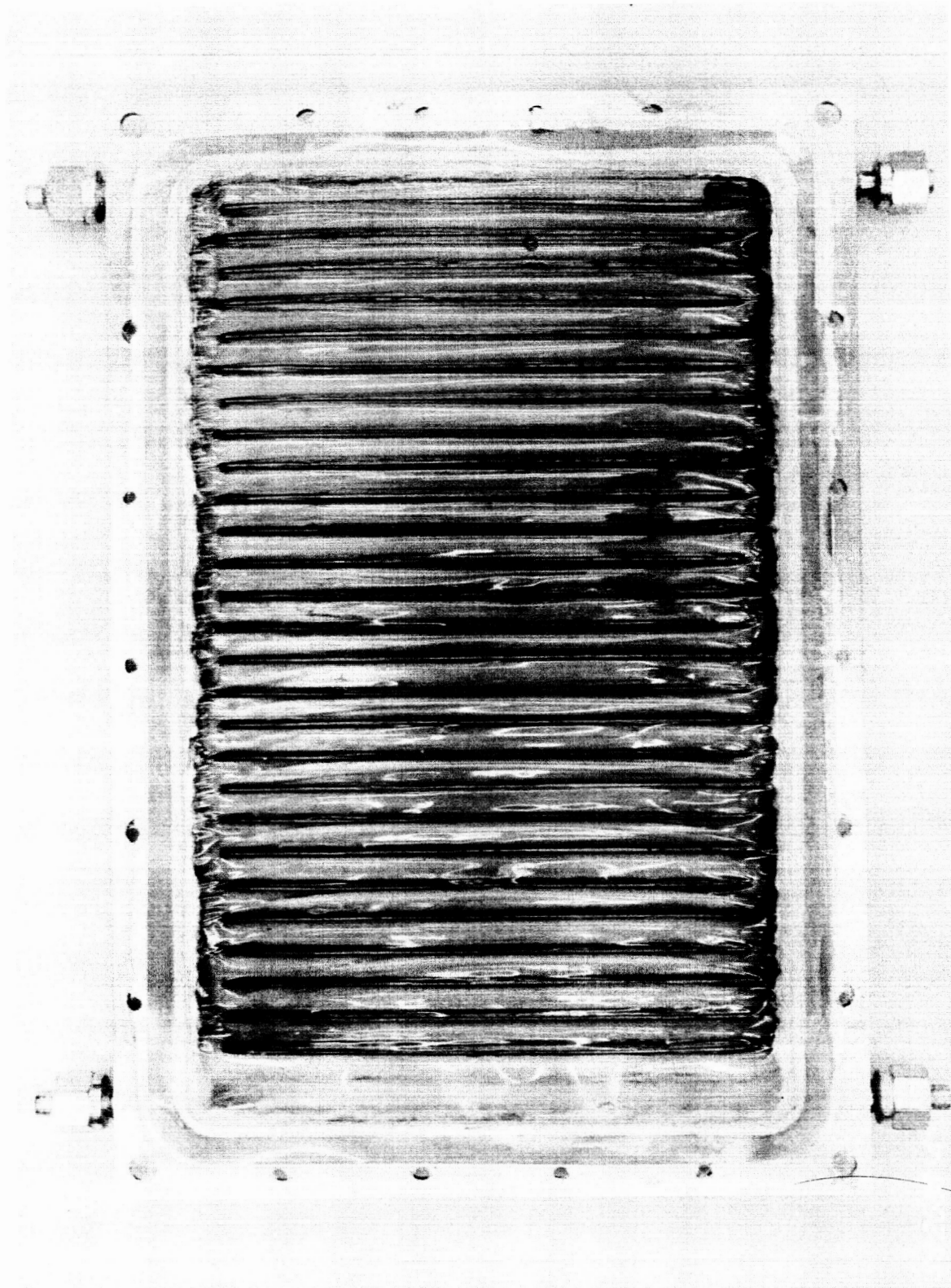


FIGURE 35. ACCEPTANCE TEST EVAPORATOR WITH MEMBRANE
AT TEST CONCLUSION

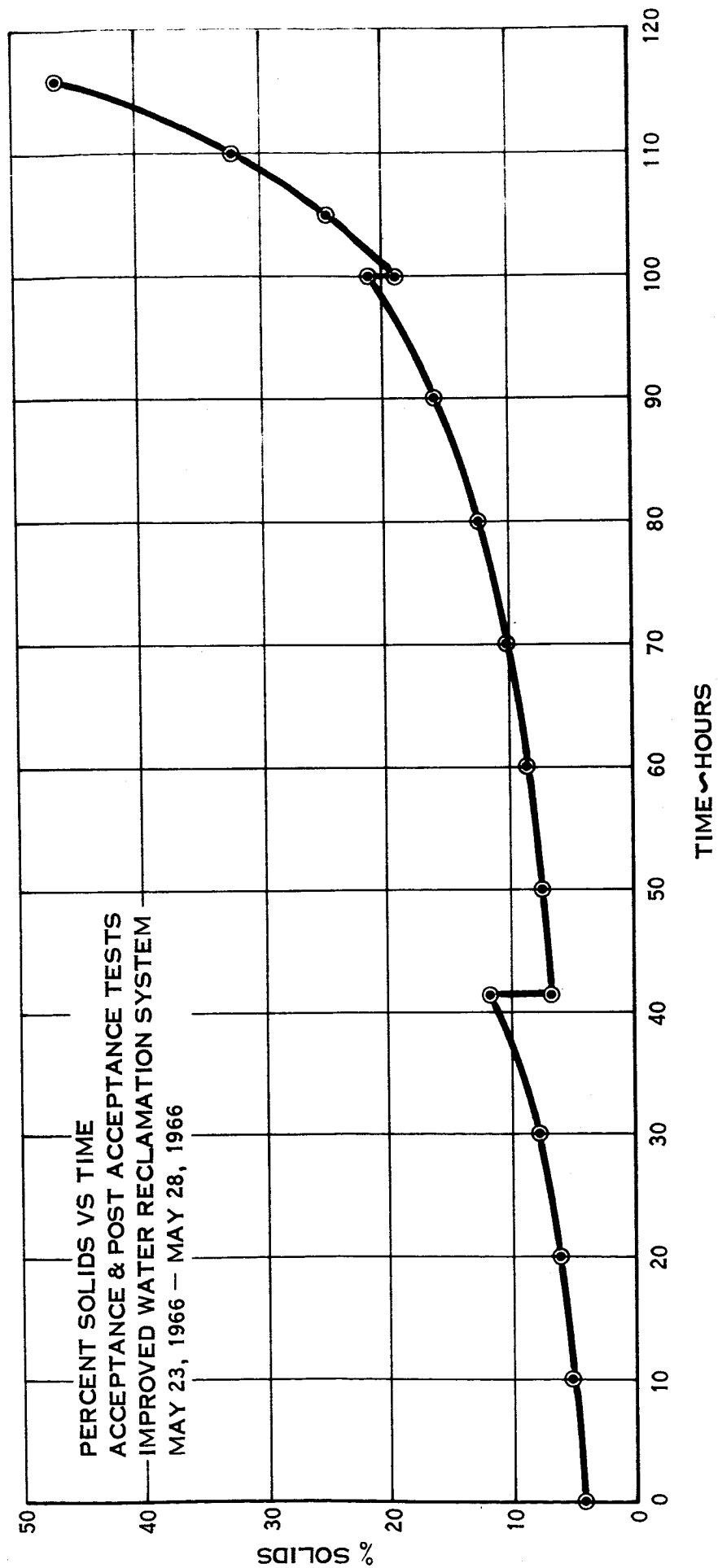


FIGURE 36. BULK BRINE SOLIDS FRACTION AS A FUNCTION OF ACCEPTANCE TEST DURATION

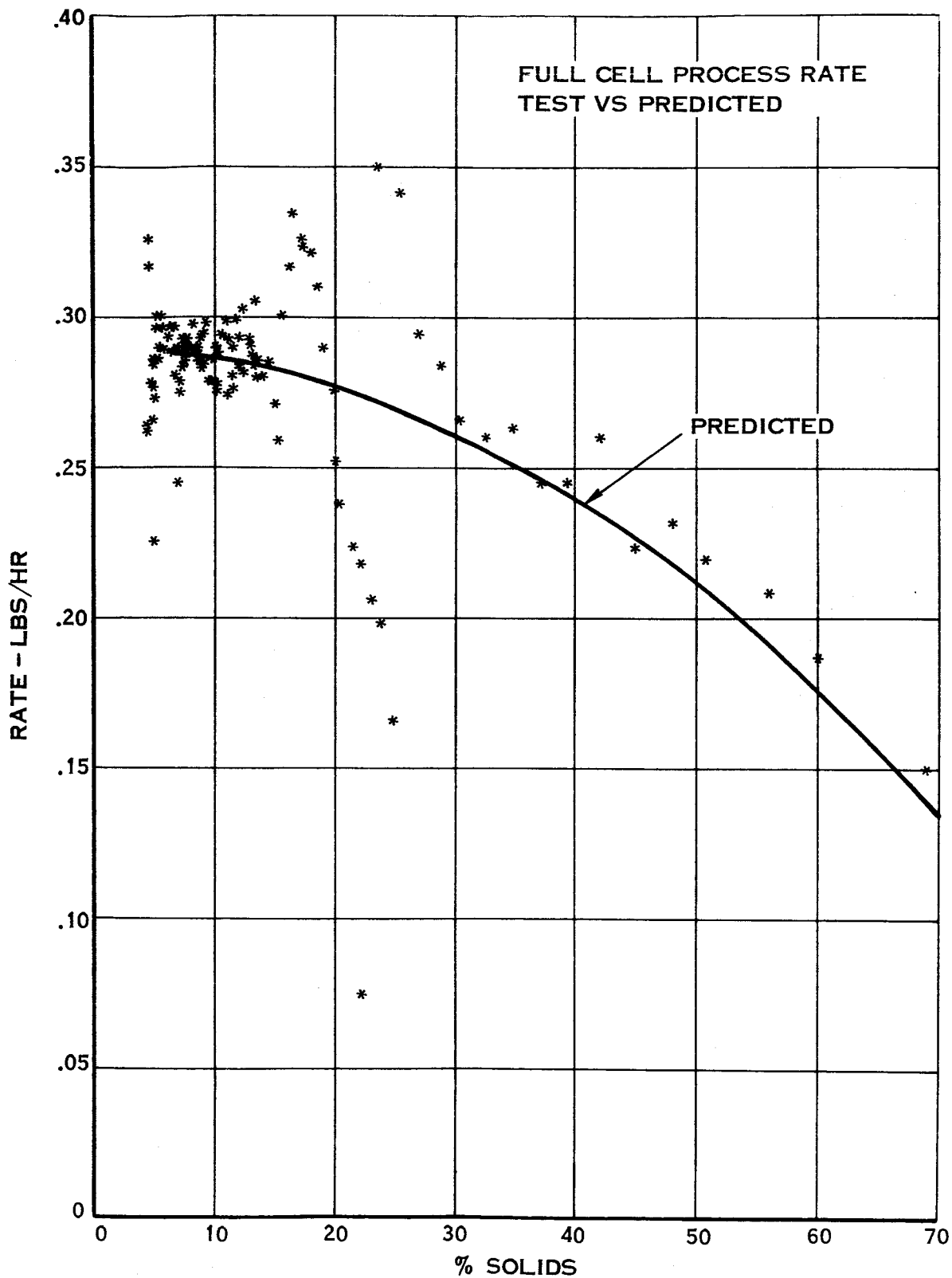


FIGURE 37. DIFFUSION CELL PROCESS RATE AS A FUNCTION
OF SOLIDS FRACTION FROM ACCEPTANCE TEST DATA

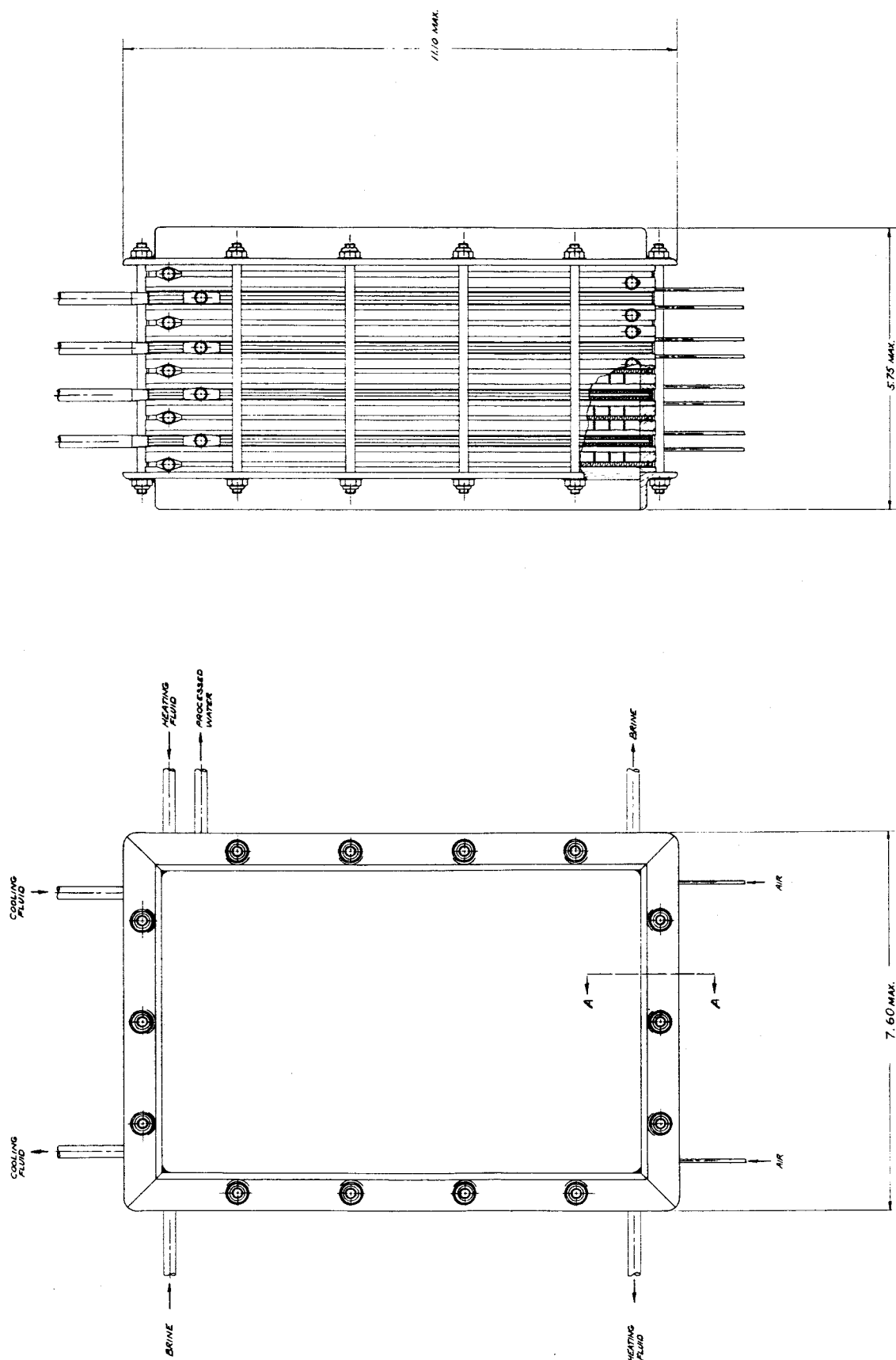


FIGURE 38. PROTOTYPE DIFFUSION STILL MODULE ASSEMBLY

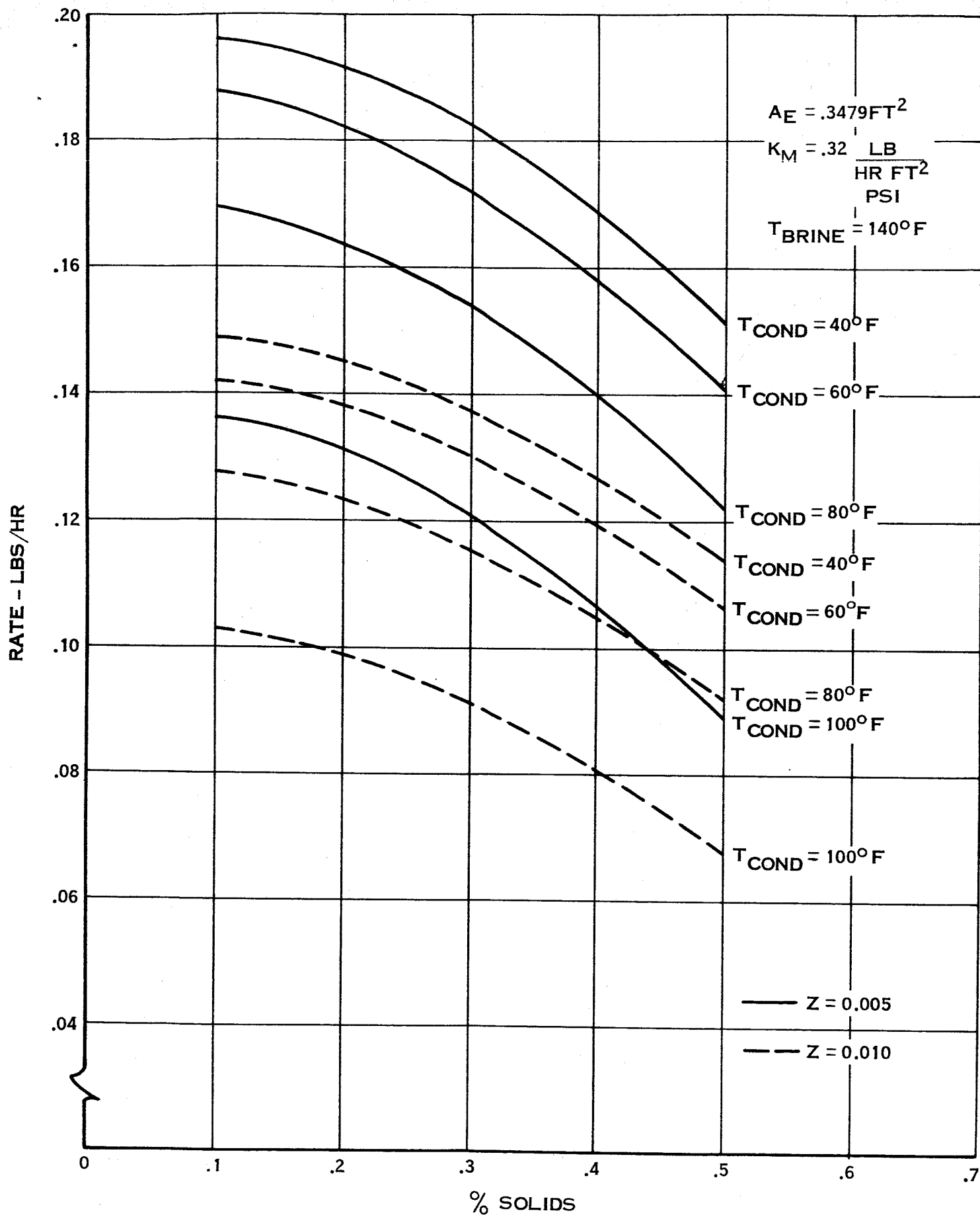


FIGURE 39. PREDICTED PROTOTYPE PROCESS RATE PER HALF CELL AS A
 FUNCTION OF CONDENSING TEMPERATURE, DIFFUSION GAP
 AND BRINE SOLIDS FRACTION

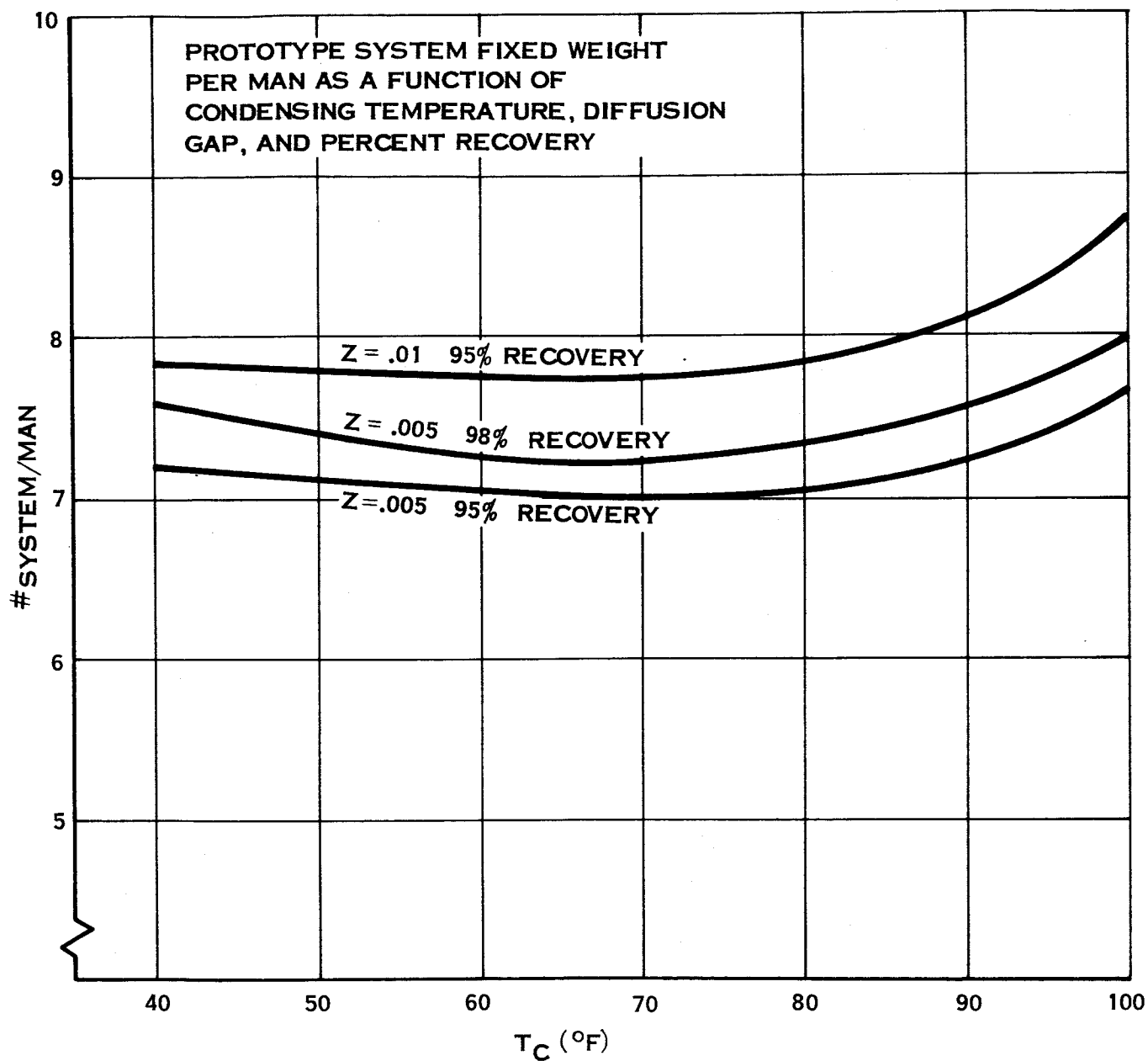


FIGURE 40. PROTOTYPE SYSTEM FIXED WEIGHT PER MAN AS A FUNCTION OF CONDENSING TEMPERATURE, DIFFUSION GAP AND PERCENT RECOVERY

PROTOTYPE SYSTEM LAUNCH WEIGHT PER MAN
AS A FUNCTION OF PERCENT RECOVERY
AND MISSION DURATION

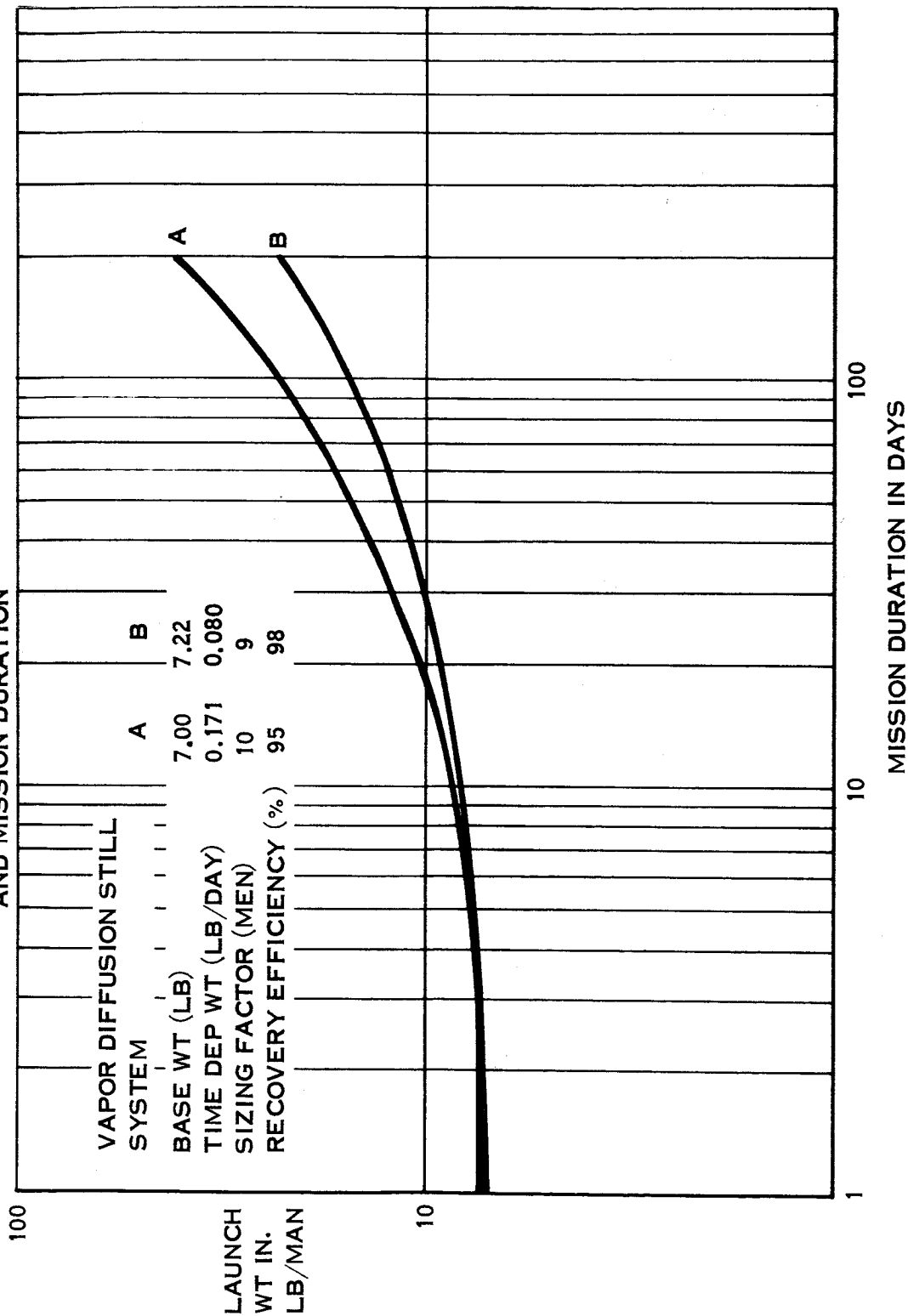


FIGURE 41. PROTOTYPE SYSTEM LAUNCH WEIGHT PER MAN AS A FUNCTION OF PERCENT RECOVERY AND MISSION DURATION

REFERENCES

1. Putnam, D. F., "Diffusion Still Feasibility Test Data", Hamilton Standard Internal Correspondence PR #135, Windsor Locks, Connecticut, July, 1965.
2. Mott Metallurgical Corporation, An Engineering Handbook on Controlled Porosity for Precision Products, Hartford, Connecticut.
3. Doty, P. M., Aiken, Captain Wm. H., and Mark, H., "Water Vapor Permeability of Organic Films", Department of Chemistry, Polytechnic Institute of Brooklyn New York, November, 1944.
4. E. I. DuPont De Nemours and Company, Inc., Bulletin C-1B #A-39158, "Technical Information, DuPont Cellophane", Wilmington, Delaware.
5. United States Department of the Interior, Office of Saline Water, Research and Development Progress Report No. 111, "Reverse Osmosis for Water Desalination", May, 1964, Chapter VII.
6. Reid, C. E. and Spencer, H. G., "Ultrafiltration of Salt Solutions at High Pressures", Department of Chemistry, University of Florida, Gainesville, Florida, April, 1960.
7. Warner, A. W., Brown, D. L., and Glass, W., "Recovery of Potable Water From Urine by Membrane Permeation", Ionics Incorporated Report No. AMRL-TDR-64-73, Watertown, Massachusetts, September, 1964.
8. Putnam, D. F., "Vapor Pressure and Other Physical Properties of Concentrated Human Urine", Hamilton Standard Report No. HSER 3768, Windsor Locks, Connecticut, November, 1965.
9. Putnam, D. F., "Chemical Aspects of Urine Distillation", ASME Paper No. 65-AV-24, The American Society of Mechanical Engineers, New York, N.Y., January, 1965.
10. McBain, J. W., and Kistler, S.S., "Membranes for High Pressure Ultrafiltration", Trans. Faraday Soc., V 26, #107, pt. 4, 1930, pp. 157-62.
11. Heuser, Emil, The Chemistry of Cellulose, p. 57.
12. Coe, W. and Allen, J., "Membrane Feasibility Testing", Hamilton Standard Report No. SVHSER 2508, Windsor Locks, Connecticut, (1966).
13. Putnam, D.F., "Experimental Mass Transfer Coefficients for Evaporation and Condensation of Water Vapor", Hamilton Standard Report No. AD-70, Hamilton Standard Division, Windsor Locks, Connecticut, 1965.

REFERENCES (Continued)

14. Proprietary Product of Minnesota Mining and Manufacturing Company, St. Paul Minnesota, "Personal Communication".
15. Runchey, F., "Operation of Water Expulsion Device with Wetting Agent Additive", Hamilton Standard Report No. HSIR 2269, Supplement A, Windsor Locks, Connecticut, February, 1965.
16. U.S. Department of Health, Education and Welfare, "Public Health Service Drinking Water Standards", Washington, 25, D.C., 1962.
17. Dushman, Saul, Scientific Foundations of Vacuum Technique, John Wiley and Sons, Inc., New York, New York, 2nd Edition, August, 1962, Chapter 7.
18. Filby, E. and Maass, O., "The Sorption of Water Vapor on Cellulosic Materials", Canadian Journal of Research, Volume 13, Section B, The National Research Council of Canada, July, 1935, 11. 1-10.
19. Urquhart and Williams, J. Textile Inst., 20, T126, 1929.
20. McBain, J. W. and Stuewer, R. F., Journal Physical Chemistry, 40, 1157 (1936).
21. Webb Associates, "NASA Life Sciences Data Book", First Edition, Yellow Springs, Ohio, June, 1962.
22. Collins, V. G. and Popma, D. C., "Water Reclamation and Conservation in a Closed Ecological System", Paper presented at the Ecological Technology Symposium, Washington, D. C., February, 1966.